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SUPERCRITICAL CARBON DIOXIDE INTERACTION WITH POLYMERIC MATERIALS: DESORPTION DIFFUSION COEFFICIENT MEASUREMENTS



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14. ABSTRACT

Chemical-polymer compatibility is an important criterion in the development of solvent-based extraction and cleaning systems for removal of toxic contaminants from polymeric surfaces. The potential degradation of polymer properties and performance is especially critical for sensitive equipment cleaning, decontamination, and extraction. The candidate cleaning fluids for sensitive equipment include fluorocarbons and supercritical fluids. An overall material evaluation strategy has been developed for evaluating the degradation of polymer properties by exposure to supercritical fluids. The initial evaluation involves the measurement of the sorption and desorption diffusion coefficients in a spectrum of polymer compositions. The objective was to characterize the complete desorption process by the continuous measurement of the entire diffusion process. Another purpose of diffusion coefficient measurements was to provide an estimate of the time required for each material to completely desorb all of the carbon dioxide cleaning fluid and return the material to the original unplasticized condition. These measurements and calculations provided the high data density required to calculate a low concentration Fick's Law diffusion coefficient and equation for the extrapolations. The composition spectrum selected contains hydrogen bond donor, hydrogen bond acceptor, dipolar, and non-polar polymer structures. Over 20 thermoplastics and elastomers were studied with supercritical carbon dioxide.

15. SUBJECT TERMS

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PREFACE

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SUPERCRITICAL CARBON DIOXIDE INTERACTION WITH POLYMERIC MATERIALS: DESORPTION DIFFUSION COEFFICIENT MEASUREMENTS

1. INTRODUCTION

Chemical-material compatibility is a critical criterion in development of a decontamination system. The degradation of material properties and performance is especially important for sensitive equipment cleaning and decontamination. Sensitive equipment includes electronics, optics, and other high-value and unique equipment. The candidate cleaning fluids for sensitive equipment include supercritical carbon dioxide and fluorocarbons.

The overall material evaluation methodology for compatibility with decontaminants is under development.¹⁻³ The selection of representative materials for compatibility testing is underway based on several parallel approaches. These include a top-down, material-to-material analysis that identifies surface materials in a spectrum of high-cost equipment. Several material science based approaches were also applied to identify a spectrum of material physical states, molecular structures, and hydrogen bond and polarity classes.⁴

The initial evaluation in any chemical-material evaluation test scheme usually involves measurement of sorption or solubility of the candidate cleaning fluid in a spectrum of relevant material compositions. This methodology can also be applied to the desorption measurements of the contaminants by a thermal decontamination process or by solvent system based decontaminants, such as Decon Green. The desorption diffusion methodology developed is also applicable to investigations on the environmental fate of hazardous chemicals on material surfaces in wind tunnel experiments. The supercritical carbon dioxide processing conditions and material sorption experimentation are being reported separately. The experimentation reported herein concentrates on the desorption diffusion kinetic process for supercritical carbon dioxide from polymeric materials.⁵

The purpose is to characterize the complete desorption diffusion process by continuous measurement of the entire diffusion curve, with the exception of a few minute time increment at the start of the experiment and a few percent of the final weight-loss. The next purpose was to use the continuous desorption curve to provide an accurate extrapolation of the zero-time sorption value immediately after supercritical carbon dioxide decompression. This extrapolation from the high concentration region of the desorption curve allowed calculation of a corrected sorption value for each material that provided an accurate ranking of material solubility relative to the uncorrected values. The characterization of the plateau to equilibrium desorption allowed the measurement of extractables and, therefore, the correction of the sorption value for this fraction extracted. Another purpose of diffusion coefficient measurements was to provide an estimate of the time required for each material to completely desorb all of the carbon dioxide cleaning fluid and return the material to the original unplasticized condition. These measurements and calculations provide an estimated 'time before reuse' after the decontamination process. The continuous measurement of desorption provided the high data density required to calculate a low concentration Fick's Law diffusion coefficient and equation

for the extrapolation to initial material sample weight before exposure. The experimental strategy includes mechanical, optical, thermal, and electrical property characterization to measure the influence of the sorbed decontaminant. In this initial screening stage, indentation hardness measurements of surface mechanical properties were completed.

2. EXPERIMENTATION

2.1 <u>Extraction Instrumentation.</u>

The extraction instruments and cells employed to expose the materials to supercritical carbon dioxide are listed in Table 1. The instrument cell nomenclature, dimensions, and volume are provided in the first four columns. The dimensions refer to the useable space for material specimens. The fifth column lists the type of test performed with specimens exposed in the various cell sizes. In general, the diffusion specimens were relatively small; therefore, the smaller cell sizes were employed.

Table 1. Supercritical Fluid Cell Dimensions and Volume for Extraction Equipment of Several Different Capacities

		Dimensions			
Cell	Equipment	(cm): diameter x length	Volume (mL)	Test Specimen	Materials: Cell with Specimen Packing
1	Hewlett Packard Cartridge (07680- 61370) for HP7680T	1 x 7	5.5	Desorption	Stainless steel with Whatman GF/A 90mm glass microfibre filter
2	Applied Separations Cell for Applied Separations SCF Extractor	1.3 x 15	24	Desorption, Flex, DMA	Stainless steel with Applied Separations Polypropylene Wool
3	Thar Designs Cell for Applied Separations SCF Extractor	3 x 23	140	Tensile, thermoplastics	Stainless steel
4	Thar Designs Cell for Applied Separations SCF Extractor	5.3 x 15	350	Tensile, elastomers	Stainless steel
5	Vessel for CF Technologies Extractor	17 x 43 basket	6500	Tensile, others	Stainless steel

2.2 <u>Desorption Instrumentation</u>.

A Mettler Model AT261 (Toledo, OH) macrobalance balance was used for correlation of specimen weights with the microbalance and provided reproducibility of 0.015 mg and readability of 0.01 mg. A New Age Industries Model Exacta EX200/300 Series instrument (New Age Industries Inc., Willow Grove, PA) was used for the indentation hardness measurements.

The thermogravimetric instruments employed to measure the continuous desorption of sorbed supercritical carbon dioxide were a TA Model TGA 2950 and Model TGA 951 (TA Instruments Inc., New Castle, DE). The final values reported were all measured with a combination of the Model TGA 2950 and control software Version 8.10B (2.3A).

Custom specimen holders were required for the unique research measurements and were constructed to clamp the ca. 1-cm diameter disk shaped specimens, allowing free surface diffusion on all sides. The 0.62-0.64 mm diameter nichrome wire was formed to a shape that matched the TGA Model 2950 hang down wire hook, cleared the furnace sides, and avoided buoyancy fluctuations.

The experimental conditions employed in the desorption measurement process are recorded in Table 2. The microbalance performance depends on the range and capacity used in the experiment, and these are listed in the first three rows of the table. The resolution and accuracy of the desorption measurements are listed next, based on the microbalance specifications for the range employed. The data acquisition rate of 6 points/min was more than adequate for the slow desorption process. Slower rates were employed for data segments near the plateau region, as noted in the data set documentation. The desorption temperature of 30 °C was selected to provide a temperature near environmental temperatures that could be effectively controlled due to the offset from ambient temperatures of near 25 °C. The flow rate setting was high enough to avoid back condensation and avoided buoyancy effects. The pre-equilibration time was the time required to equilibrate the specimen chamber at 30° so there was a minimum loss of temperature during the 2 min required to open the furnace, load the specimen, and close the furnace. A temperature-programmed ramp to 30° minimized temperature overshoot as the furnace chamber re-equilibrated at 30° after the specimen was mounted on the wired holder within the chamber.

2.3 Chemicals and Materials.

The supercritical carbon dioxide and polymeric materials used are documented in Tables 3-5.

Several categories of material samples were studied in the initial experimentation. One category was based on a survey of materials in sensitive equipment. Another category included thermoplastics tested in previous supercritical carbon dioxide studies to compare the reproducibility of the material sorption process in our laboratory with published data sets. The exact samples employed by others could not be obtained; therefore, better-documented analogs were procured from other sources. Another set of materials was selected from a set of reference materials developed for chemical defense testing. The polymer compositions were those from chemical defense protective equipment that would be brought to and worn at contaminated

sensitive equipment decontamination sites. Materials used in protective masks and gloves were emphasized. The material sources, the codes used to label the material samples, and the source addresses are listed in Table 4. Each material sample source is documented in other tables based on these codes. The itemized sample number is listed, followed by the complete nomenclature, the code, the thickness, and the company source code (see Table 5). Some of the materials in Table 5 were only employed in sorption screening experiments, and detailed desorption diffusion coefficient measurements were not performed on these materials. The subset of the materials with diffusion measurement values are highlighted in Table 5, and the data are listed in the tables in the results section.

Table 2. Experimental Conditions and Specifications Employed for the Desorption Diffusion Coefficient Determination

Variable	Value
Range	0.1 µg to 100 mg
Capacity, total load	5 g
Capacity, weighing	1.0 g
Resolution	0.1 µg
Accuracy	± 0.1%
Data Acquisition rate	6 points/min for the 1st 24 hr; 1 point/min after 24 hr
Temperature, °C	30.00
Flow rate, cc/min	100 (60 + 40)
Pre-equilibration time	> 2 min
Temperature programmed ramp to isothermal	2º/min

Table 3. Carbon Dioxide Sources for Supercritical Experimentation

Instrument	Applied Separations SCG Extractor	Hewlett Packard 7680T
Grade	"Bone-dry"	Supercritical Fluid Extraction, SFE
Purity, %	99.8 minimum	99.99 minimum
Valve	CGA 320	CGA 320
Source	Roberts Oxygen Co., Rockville, MD 20855	BOC Gases, Rivertown, NJ

Table 4. Sources of the Material Specimens Employed in Desorption Experimentation

Material Source	Code	Address
Goodfellow, Inc.	GF	800 Lancaster Ave; Berwyn, PA 19312-1780; (800) 821-2870
Harbor City Plastics	HC	1306 Governor Court; Abingdon, MD 21009
Smithers Scientific Corp.	SS	Smithers Scientific Services, Inc.; 425 West Market Street, Akron, OH 44303-2099; (330) 762-7441 Fax: (330) 762-7447
Resin Kit	RK	1112 River St; POB 509; Woonsocket, RI 02895-0509

Table 5. Polymeric Compositions Employed in Supercritical Carbon Dioxide Interaction Experiments

No.	Polymer Name	Polymer	Nominal Thickness mils	Source Code
1.	Polyethylene, High Density	HDPE		GF
2.	Polyethylene, High MW	HMWPE		GF
3.	Polyethylene, Low Density	LDPE		GF
4.	Polyethylene-propylene	PEP	49	GF
5.	Polytetrafluoroethylene	PTFE		GF
6.	Poly(0.9-styrene-co-0.1-butadiene) Polysar 3350, high impact	PSB	75	
7.	Polyvinylidenefluoride, Kynar	PVDF		GF
8.	Polyvinylchloride, unplasticized	UPVC		GF
9.	Polysulfone Thermalux	PSF		GF
10.	Polyvinylchloride, plasticized	PVC	41	HC
11.	Poly(2,6-dimethyleneoxide) Noryl	PPO	34	HC
12.	Polycarbonate Lexan	PC	35	HC
13.	Polymethylmethacrylate Plexiglas	PMMA	39	HC
14.	Acrylonitrile butadiene styrene (20-8-72%) Royalite	ABS	62	GF
15.	Poly(imide) film	PIF	11	GF
16.	Polyetheretherketone	PEEK	20	GF
17.	Polyurethane	PÜ	42	GF
18.	Poly(oxymethylene) Delrin	POM		GF
19.	Polyethylene terephthalate Mylar	PET		GF
20.	Cellulose –OH) acetate butyrate Uvex	CAB		GF
21.	Polyethylene terephthalate glycol, Vivak	PETG		GF
22.	Polyetherimide Ultem	PEI		GF
23.	Polyamide Nylon66	PA		GF
24.	Poly(isoprene) Natural Rubber	PIP	41	SS
25.	Poly(isobutylene) Butyl Rubber	PIB	41	SS
26.	Poly(bromobutylene) Bromobutyl Rubber	PBB	42	SS
27.	Poly(dimethylsiloxane)	PDMS	47	SS
28.	Silicone Modified Organic polymer (proprietary)	SMO	75	proprietary
29.	Poly(styrene-butadiene) rubber	PSBR	41	RK
30.	Poly(tetrafluoroethylenepropylene) Aflas	PTFEP	38	SS

2.4 <u>Procedures</u>.

2.4.1 <u>Supercritical Exposure Procedures.</u>

The supercritical exposure procedures and conditions employed were based on conditions that were found to successfully remove contaminants from non-sorptive surfaces in previous exploratory experiments (B. MacIver, D. Sorrick; SBCCOM). The supercritical conditions are documented in Table 6. The extraction equipment and cells are documented earlier in this document (see Section 2.1).

Table 6. Comparison of Experimental Conditions for Supercritical Carbon Dioxide Sorption in Polymeric Materials: Measurement Conditions versus Similar Literature⁶⁻⁸
Conditions

Experimental Conditions	Units	RT#1	RT#2	Literature C3
Pressure	Psig.	1399	1399	2000
Temperature	°, C	50	50	40
Time: Static Extraction @ PT	Min	15	15	60
Time: Decompression	Min	1	1	60
Time: 1 st Sorption Weighing	Min	5	20	20

The first column in Table 6 lists the supercritical pressure, temperature, and exposure-time conditions. The second column documents the units. The remaining columns define codes that document the set of experimental conditions in the table. These codes are used in experimental data sheets and computer data file documentation. The difference in RT#1 and RT#2 is only the sorption weighing time; this weighing time at 20 min allows a direct comparison with literature (last column) that reported the first weighing at 20 min.

The extraction time refers to a static exposure without flow through the cell. The decompression time is a rapid but controlled pressure drop to ambient at an approximately linear rate. The "Sorption Weighing" time defines the time lag required to disassemble the cell and manipulate the material specimen to obtain the initial weight. Carbon dioxide desorption under ambient conditions occurred during this lag and can be corrected by an extrapolation back to the end of the decompression period.

2.4.2 <u>Sorption Procedures</u>.

The procedures for exposing the material samples to the supercritical extraction and cleaning process have been documented separately; therefore, these procedures are briefly surveyed in Table 7.

2.4.3 <u>Desorption Procedures.</u>

The procedures for the desorption measurement involve an interleaving of specimen manipulation with software control steps, therefore, these are documented in detail in the Appendix A. A general survey is provided in Table 8.

2.4.4 <u>Kinetic Analysis Data Reduction Procedures.</u>

The basic data sets consisted of time, temperature, and weight measurements. These files were processed by various filtering techniques and data conversions to Fickian parameters. Interim results were processed with custom C++ code based on our previous operational computational methods. A general description of the kinetic data analysis procedure is provided in Table 9. The final computations were performed using custom coded Excel functions, and the documentation is provided in Table 10.

Table 7. Procedures for Supercritical Sorption of Polymeric Materials

Thermoplastic and elastomer sheet specimens were stamped out using 3/8-inch (or other size) hollow punch set (SE Corp.)

Thermoplastic molded specimens were used "as is".

Specimens were pre-weighed, labeled and placed in individual containers.

A set of 3-6 specimens was loaded into a supercritical cell with separation by Whatman GF/A 90mm glass microfibre filter packing sheets for desorption disk specimens and separation by Applied Sciences Polypropylene Wool for other specimen sizes.

Specimen identity was retained by a first-in/first-out sequencing.

Supercritical instrument controls were set for the instrument model employed.

A loaded cell was inserted or connected and the experiment was initiated.

A laboratory stopwatch was started at the end of the decompression step, when the specimen returned to atmospheric pressure.

The supercritical cell was rapidly disassembled in order to weigh the specimen within 4-5 min of the end of the decompression step.

The specimen weight and time after decompression were recorded in a hardcopy data capture version of the spreadsheet.

Specimens were submitted to post-exposure experimentation and the time after decompression was recorded from the stopwatch, for example, diffusion coefficient measurements by TGA (or potentially, for glass transition by DMTA or mechanical properties by Universal Test Machine).

Table 8. Procedures for Desorption Diffusion Coefficient Determination by Thermogravimetric Instrumentation

Standard calibration procedures were completed according to the operations manual

The pre-exposed specimen was weighed on the TGA at 30 °C, removed, and submitted for exposure.

The TGA was programmed to pre-equilibrate at 30° for at least 2 min.

All possible pre-experiment controls were set to minimize the time between loading the desorbing specimen and the start of data acquisition.

After the specimen was removed from the exposure cell, the TGA furnace was opened, the specimen mounted, the furnace closed, and data acquisition initiated.

Stopwatch time was recorded in the TGA remarks field to document the lag time between the end of decompression and the first desorption data point.

Real-time plotting was initiated to allow an estimation of the attainment of a plateau and the presence of extraction by the mass signal falling below the initial mass.

After a plateau was attained, the experiment was terminated, the specimen was transferred from the TGA microbalance to the macrobalance and comparative mass was recorded.

Macrobalance recordings were completed as needed to check for further desorption.

DMTA or other property measurements were performed as a function of specimen weight and desorption time (not reported herein).

Indentation hardness was measured after desorption was complete and compared with values measured on unexposed specimens from the same lot.

Table 9. Kinetic Data Analysis Procedures for Desorption Diffusion Coefficient Calculations

Material identification and the sorption and desorption conditions were stripped from the file headers into the data sheet formats.

Additional material and sorption/desorption experiment documentation were keyed into the data form.

Pre-exposure data and statistics were calculated by the code or internal instrument functions for the diameter, thickness, mass, and indentation hardness.

Exposure data and statistics were calculated: Exposure time, time lag, mean temperature with 95% confidence interval, sorption weight, sorption weight %, sorption weight corrected for extractables, sorption weight % corrected for extractables, indentation hardness with percent change.

Time-temperature-mass data sets were reduced to kinetic parameters by: converting units; converting mass to relative mass; converting lapsed time to absolute time after end-of-decompression; converting time to Fick's Law time^(1/2).

Converted data sets were regressed using a Fick's Law relation of Reduced-mass versus Time^(1/2) over two regions: High Concentration Region and Low Concentration Region. The high concentration region in the desorption experiment contains the initial diffusion from the swollen polymer surface, not meeting Fick's Law criteria; the low concentration region contains the final diffusion from the depleted and unswollen polymer surface, meeting Fick's Law criteria.

The procedure for selecting the range for the high concentration region consisted of including the coordinate for zero-time and 1.0 Reduced Mass, the initial data acquisition point, then through to the inflection point for deviation from the linear regression fit, usually occurring over a reduced mass range of about 0.65 to 0.55.

The regression was fitted through a single initial zero-time versus 1.0 Reduced Mass coordinate on the y-axis and contained an initial region without data points due to the time lag between decompression and the start of the desorption data acquisition.

The procedure for selecting the range for the low concentration region consisted of including the coordinate for the last data acquisition point through to the inflection point for deviation from the linear regression fit, usually occurring over a reduced mass range of about 0.08 to 0.02.

The low concentration plateau was classified into one of three observed cases and appropriate data reduction procedures were applied for each case, as follows.

Case 1: The plateau occurred near the initial mass and therefore, the absolute and relative reduced masses were similar. The final mass was defined as the initial zero-time mass.

Case 2: The plateau occurred below the baseline for initial mass, indicating extraction had occurred. The last data point was assigned as the final mass for defining the total mass of liquid sorbed and desorbed.

Case 3: The plateau occurred significantly above the initial mass and could not be extrapolated to the initial mass axis in a reasonable time. The final mass was defined as the initial, zero-time mass. (This data reduction procedure ignores the possibility of a tare off-set, however, for the small differences observed between the plateau mass and initial mass, the effect on the results was small)

The low concentration diffusion coefficient equation was used to calculate the time to complete desorption.

Table 10. Equations Employed for Sorption and Desorption Diffusion Data Reduction and Analysis

and Analysis	
Cell Number or Cell Range,	
EXCEL	Documentation
An: File transfer from instrument	Time, min
Bn: File transfer from instrument	Temp, degrees celcius
Cn: File transfer from instrument	TGA mass, mg
Dn = (An * 60)	Converting time in min (An) to time in seconds (Dn)
En = (Dn + M4)	Adjustment to time for extrapolation to "zero" time
Fn = (En / 3600)	Converting adjusted time in seconds (En) to hours (Fn)
Gn = SQRT (En)	Function for Fick's Law square root of time dependence: En^1/2>Gn column
Hn = (((Cn - K8) / K8) * 100)	Converting mass remaining in milligrams to percent
, , ,	using the initial, dry sample mass as the reference mass
In = ((Cn - K8) / (K4 - K8))	Fractional mass desorbed: Column I created from
Y = (1 (7/4 C=) / (7/4 149))	Column C
$J_n = (1 - ((K4 - C_n) / (K4 - M8)))$	Fractional mass remaining: Column J created from Column C
K4: Cell transfer or from	Extrapolated mass at zero time
ANALIZ file	
K8: Keyed input or from ANALIZ	TGA initial, dry mass
file	
K12 = PI() * POWER (((N20 *	Computing High Concentration Diffusion Coefficient
P20)/4),2)	(HCDC)
K15: Cell transfer	R-Square for regression equation for HCDC calculation
K18 = (-P19) / P20	X-intercept for HCDC
K21: Cell transfer	Y-intercept for regression equation for HCDC
	calculation
K25 = P19 * K4	Converting y-intercept for HCDC into mass, mg.
L4 = (((K4 - K8) / K8) * 100)	Converting "zero" time mass to wt. %
L8: Cell transfer	Equilibrium mass of desorbed sample, at end of
	experiment
L12 = PI() * POWER (((N20 *	Computing Low Concentration Diffusion Coefficient
P43)/4),2)	(LCDC)
L15: Cell transfer	R-Square for regression equation for LCDC calculation
L18 = (-P42) / P43	X-intercept for LCDC
L21: Cell transfer	Y-intercept for regression equation for LCDC calculation
L25 = (POWER (L18,2)) / 3600	Converting x-intercept for LCDC into time, hrs.
M4: Keyed input or from	Adjustment to account for elapsed time in seconds
ANALIZ file	between sample exposure and start of experiment after
	sample loading
M8: Keyed input	Minimum mass used for LCDC calculation
M12: Cell transfer	Total mass of liquid sorbed
M25 = (POWER (L18,2)) / 600	Converting x-intercept for LCDC into time, min.

Table 10. Equations Employed for Sorption and Desorption Diffusion Data Reduction

and Analysis (Continued)

Cell Number or Cell Range,	
EXCEL	Documentation
M29: Keyed input	Maximum TGA mass from original TGA data file
M33: Cell transfer	Mass of liquid sorbed based on maximum TGA mass in M29
M37= (M33/K8)/100	Sorption, percent, based on maximum TGA mass in M29
M41 = (((M37 - L4) / L4) * 100)	Relative error, %, of initial 7-12 min sorption value
N4: Keyed input or from ANALIZ file	Shore hardness scale
N8: Keyed input or from ANALIZ file	Pre exposure hardness
N12: Keyed input or from ANALIZ file	Post desorption hardness at time
N16 = (((N12 - N8) / N8) * 100)	Change in hardness, %, after exposure and desorption
N20: Cell transfer	Mean thickness, cm.
N22: Copy-paste input	Original TGA data filename
N25: Cell transfer	X-intercept for LCDC as a time in min
N29: Keyed input	Adjusted TGA mass for 20 min elapsed time from exposure
N33: Cell transfer	Mass of liquid sorbed based on 20 min elapsed time in N29
N37 = (N33/K8) * 100	Sorption, %, based on N33
N41 = (((N37 - L4) / L4) * 100)	Relative error, %, of 20 min literature sorption value
P19: Cell transfer from regression cell	Y-Intercept Diffusion Coefficient from HCDC regression equation
P20: Cell transfer from regression cell	Slope from regression equation for HCDC calculation
P42: Cell transfer from regression cell	Y-intercept Diffusion Coefficient from LCDC regression equation
P43: Cell transfer from regression cell	Slope from regression equation for LCDC calculation

3. **RESULTS**

3.1 Supercritical Carbon Dioxide Sorption and Desorption Data for Polymeric Materials.

An example of a plot of time versus desorption diffusion mass loss is shown in Figure 1. In this example for poly(styrene-butadiene) (PSB) thermoplastic, the desorption process is well behaved, and the carbon dioxide loss plateau is approaching the zero-time baseline. The full compilation of data sets is provided in Appendix B. Subsets of the data are organized in tables in the following section to allow an evaluation of trends in the data as a function of experimental and data reduction techniques.

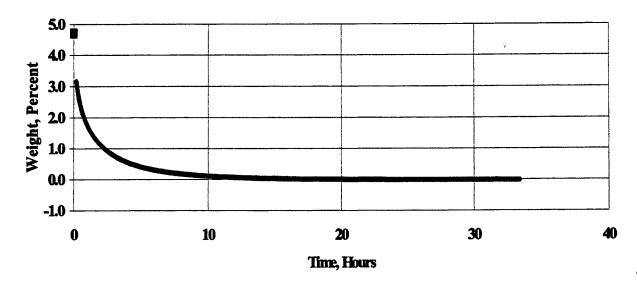


Figure 1. Desorption Diffusion (30 °C) of Supercritical Carbon Dioxide from Polymeric Materials: PSB (0140d)

3.2 <u>Sorption Values Corrected for Fraction Extracted Employing Equilibrium</u> Desorption Measurements.

The sorption results in Table 11 are corrected for the fraction of mobile additives in the material that were extracted during the supercritical exposure. Poly(isoprene), PIP in Figure 2, is an example of a polymer containing mobile additives that appear to be partially extracted; the carbon dioxide desorption plateau is below the original baseline providing a measurement of the fraction extracted. The positive 'Extracted, %' values in column 5, Table 11, denote the desorption diffusion data plateaued below the initial baseline and indicated a net extraction. The (+/-) sign convention is reversed in evaluating the raw mass data, because the mass values below the zero baseline are negative when net extraction takes place. The magnitude of the fraction extracted is programmed into the data reduction procedures to calculate the corrected sorption. The value for the corrected sorption, based on the fraction extracted (in the third column of the table), is followed by the uncorrected sorption value. The relative error due to ignoring the extractables is calculated in the last column. Note that experimentation that ignores the extraction of the additives in the material causes considerable error that would change the relative ranking of materials for resistance to supercritical carbon dioxide sorption. The 'Not Applicable' entry refers to materials in which there was no measurable extraction. The methodology was capable of ranking the relative sorption levels for the spectrum of materials. The corrected sorption value was required to correctly rank the materials. The general trend in supercritical carbon dioxide was slightly influenced by fraction extracted. For the combination of low sorption and high extractables (e.g., PEEK), the relative error was high but the ranking was only slightly influenced.

Table 11. Supercritical Carbon Dioxide Sorption (%) into Polymeric Materials Based on Correction for Fraction Extracted: Conditions of 1400 psig and 50 °C after 15 min

	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	13 mm		T	
.,	Polymer	Sorption,	Sorption,	Extracted,	Relative
No.	Codes	Corrected, %	Uncorrected, %	%	Error, %
1.	PDMS,	0.44	0.44	-0.03	Not Applicable
	139d				
2.	PIF, 0151z	1.09	0.21	0.88	-80.7
3.	PVC,	1.47	1.47	-0.03	Not Applicable
	0089d	<u></u>			1 11
4.	PEEK,	1.49	1.20	0.28	-18.8
	154z	<u> </u>			
5.	PVC,	1.65	1.65	-0.02	Not Applicable
	0110z				The state of the s
6.	PTFEP,	2.39	2.24	0.15	-6.3
	153z				
7.	PC, 0082d	2.46	2.36	0.10	-4.1
8.	PC, 0117d	2.62	2.49	0.13	-5.0
9.	PBB,	3.12	3.00	0.12	-3.8
	0138d	_		0.12	3.0
10.	PEP, 0130d	3.22	3.11	0.11	-3.4
11.	PPO,	3.64	3.64	-0.24	Not Applicable
	0091d			0.2.	140t Applicable
12.	ABS,	3.75	3.72	0.03	-0.8
	0115d			0.05	-0.0
13.	PIP, 0131d	4.12	3.39	0.73	-17.7
14.	PPO,	4.29	4.29	-0.17	Not Applicable
	0096d			""	rvotrippiioaoie
15.	PSB, 0140z	4.72	4.70	0.01	-0.2
16.	SMO,	4.94	4.94	-0.20	Not Applicable
	0129d			3,23	riotrippiiodole
17.	PMMA,	5.83	5.83	-0.90	Not Applicable
	075z			""	Trot rippineable
18.	PMMA,	6.24	6.24	-0.22	Not Applicable
	105z		· •		Tiotripphoable
19.	PU, 0124d	7.08	6.80	0.28	-4.0
20.	PSBR,	7.32	6.58	0.74	-10.1
	143z				10.1
21.	PIB, 0137d	7.88	7.88	-0.13	Not Applicable

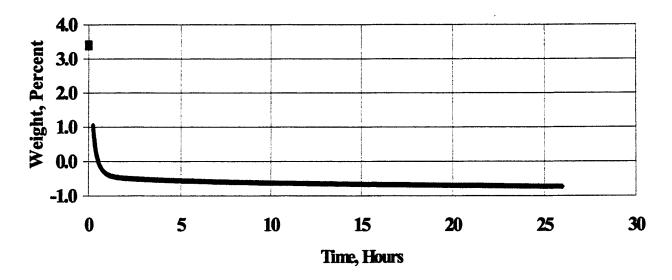


Figure 2. Desorption Diffusion (30 °C) of Supercritical Carbon Dioxide from Polymeric Materials: PIP (0131d)

Replicate specimens of PC, PVC, and PPO had reproducible 'Extractables' and 'Corrected Sorption Values' (columns 5 and 3 respectively, Table 11). The PMMA Extractible values were dissimilar, but small relative to the sorption, therefore, the rankings were identical. Note that the reproducibility and accuracy of the extractible determination is dependent on the attainment of a plateau in the continuous microbalance desorption process. Alternatively, the specimen mass could be determined after a longer period by removal from the continuous microbalance monitoring instrument and weighing on an independent macrobalance. This would also reduce the specimen residence time in the microbalance and increase specimen throughput.

About half of the materials had significant extractables under the processing conditions. Therefore, the migration of extractables cannot be ignored in the evaluation of material compatibility. The extractible adjustment is a systematic error that underestimates the true SCCO₂ sorption value. Furthermore, this error is not a constant offset for all materials and is shown here to vary considerably with polymer structure and formulation.

# 3.3 <u>Comparison of Sorption Values with Correction to Zero-time Mass Based on High Concentration Desorption Extrapolations.</u>

Most of the sorption values reported in the literature are based on an initial measurement that is often performed after considerable desorption has occurred. This desorption occurs between the time the specimen is removed from the liquid and the initial weighing; the more volatile the liquid the higher the systematic error. Supercritical carbon dioxide is an even greater experimental challenge because the true equilibrium sorption value exists at elevated temperature and pressure. The decompression period adds further uncertainty regarding the zero-time value in desorption kinetics. The end of the decompression period was arbitrarily

defined as the zero-time because we were able to hold the time-to-decompress from high to ambient pressure to a fixed 1-min interval. If the decompression period was variable, the start of the decompression process would be a better kinetic zero-time.

Figure 3 provides a Fick's Law plot of time^(1/2) versus reduced mass showing the extrapolation back to an adjusted zero-time sorption value for poly(isobutylene) (PIB). The back extrapolation for this elastomer is characteristically long, however, rapidly sorbing and desorbing polymers such as PDMS are even more extreme.

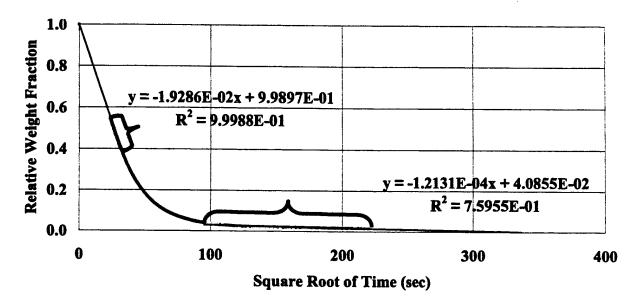


Figure 3. Fick's Law Plot of Desorption Diffusion (30 °C) of Supercritical Carbon Dioxide from Polymeric Materials: PIB (0137d)

Due to this lag before the initial sorption measurement, the traditional reported measurements give systematically low sorption values that underestimate the actual degree of interaction and solubility of fluids such as supercritical carbon dioxide in materials. In Table 12, the 'zero-time corrected sorption' values are calculated, followed by the error in ignoring the desorption during the 7 to 12-min TGA experiment and the 20-min measurement delay. Large systematic relative errors of -15 to -86% were suffered by ignoring the extrapolation to zero-time. The methodology was capable of correcting for this measurement time lag after decompression and improving the ranking the materials. One can observe from the last two columns that there would be large ranking errors and polymer-fluid interaction errors from not employing the methodology for extrapolating to the end-of-decompression. The replicates of PVC, PC, and PMMA showed good sorption reproducibility, whereas PPO replicates had a larger difference of 3.75 versus 4.3% or standard deviation of 6.7% relative.

Table 12. Comparison of Sorption (%) Corrected for Extrapolation to Zero-time at the end of the Decompression Period Versus Uncorrected Sorption after 7-12 min and 20 min (6-8)

	Polymer	Sorption, Zero-	Sorption at 7-12 min, %	Sorption at 20 min, %
No.	Codes	time Corrected	(Relative Error, %)	(Relative Error, %)
1.	PDMS, 139d	0.44	0.13 (-70.3)	0.06 (-86.8)
2.	PIF, 0151z	1.09	0.16 (-22.5)	0.04 (-80.0)
3.	PVC, 0089d	1.47	1.16 (-21.3)	1.05 (-28.8)
4.	PEEK, 154z	1.49	0.78 (-34.7)	0.60 (-50.3)
5.	PVC, 0110z	1.65	1.37 (-17.2)	1.20 (-27.2)
6.	PTFEP, 153z	2.39	1.68 (-24.7)	1.31 (-41.3)
7.	PC, 0082d	2.46	1.85 (-21.5)	1.48 (-37.1)
8.	PC, 0117D	2.62	1.85 (-25.8)	1.48 (-40.5)
9.	PBB, 0138d	3.12	1.41 (-52.8)	0.90 (-70.0)
10.	PEP, 0130d	3.22	1.53 (-50.7)	0.93 (-70.0)
11.	PPO, 0091d	3.64	2.57 (-29.5)	1.97 (-45.7)
12.	ABS, 0115d	3.75	2.86 (-23.2)	2.37 (-36.3)
13.	PIP, 0131d	4.12	1.08 (-68.2)	0.60 (-82.4)
14.	PPO, 0096d	4.29	2.74 (-36.1)	2.05 (-52.2)
15.	PSB, 0140d	4.72	3.32 (-29.3)	2.76 (-41.4)
16.	SMO, 0129d	4.94	3.92 (-20.8)	3.50 (-29.2)
17.	PMMA, 075z	5.83	4.82 (-17.3)	4.30 (-26.2)
18.	PMMA, 105z	6.24	5.26 (-15.7)	4.67 (-25.2)
19.	PU, 0124d	7.08	5.07 (-25.5)	4.19 (-38.4)
20.	PSBR, 143z	7.32	3.67 (-44.3)	1.87 (-71.6)
21.	PIB, 0137d	7.88	4.36 (-44.7)	2.72 (-65.5)

### 3.4 <u>Time for Complete Desorption and Reuse of Sensitive Equipment after Exposure</u> Employing Fick's Law Low Concentration Diffusion Equations.

It is important to establish the time required for complete desorption of all carbon dioxide from the material before equipment can be reused after decontamination. Residual decontaminant such as carbon dioxide or fluorocarbon cleaning fluids can plasticize the polymer and decrease certain properties that might cause failure under use conditions. The ability of the measurement and data reduction process to provide this type of "time for complete desorption" values is demonstrated in Table 13. The continuous measurement of desorption allowed the best possible estimate of the time to complete desorption because the high data densities provided robust extrapolations to the time required for the material specimen to regain its original weight. The approach to this final equilibrium can be extremely slow, however, and the continuous, high data densities combined with a Fick's Law diffusion coefficient allowed extrapolation to the time axis zero-mass values.

An example of a Fick's Law plot for Silicone Modified Organic (SMO) polymer is shown in the Figure 4. The mass has been normalized from 0 to 1 on the y-axis and the Fickian time^(1/2) is employed on the x-axis. The Fick's Law equations for the linear portion of

the initial and final desorption are shown adjacent to their respective section of the plot. The thinner line denotes the extrapolated section of the curve at both extremes of the plotted data. One can see the extrapolation to zero-time on the x-axis that defines the time-to-complete-desorption; this value can be used to specify the time-to-reuse for decontaminated items.

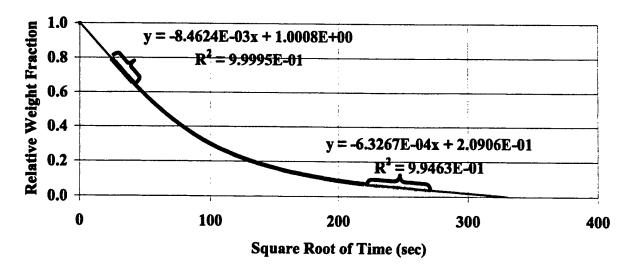


Figure 4. Fick's Law Plot of Desorption Diffusion (30 °C) of Supercritical Carbon Dioxide from Polymeric Materials: SMO (0129d)

The time for complete desorption is reported, followed by the Fickian diffusion equation and diffusion coefficient in the last two columns of Table 13. One can see that the methodology is capable of ranking the materials based on the time required to return to the unplasticized condition after super critical carbon dioxide exposure. For the materials studied, this period ranged from about 1 to 5 days.

The calculation of an extrapolated time to complete desorption can be sensitive to several processes influencing the data near the initial baseline, which are the presence of mobile extractables in the materials and the length of the experiment and degree to which the decontaminant has approached a plateau. Desorbing polymers with shorter experiment times that are not close to the baseline require longer extrapolation periods, increasing the potential error. The permutations of categories of data sets have been organized according to the following two processes:

- the low concentration desorption can be at a zero plateau or continuing to decrease toward a desorption mass change plateau
- the final mass relative to the baseline can be near zero, below zero (indicating extraction has occurred), or above zero (indicating the desorption was so slow the experiment had to be terminated before nearing complete desorption). The number of polymers in the various combinations of these two conditions have been tabulated and reported in Table 14.

Table 13. Time for the Complete Desorption of all Carbon Dioxide from Polymeric Materials Based on a Fick's Law Extrapolation of Low Concentration Desorption Diffusion

		Time for		
		Complete	Fickian Diffusion	Diffusion
	Polymer	Desorption hrs	Equation: Relative	Coefficient,
No.	Codes	(days)	Mass Fraction =	cm ² /sec
1.	PEP, 0130d	20.6 (0.9)	$0.038 - 1.38E-4 \text{ time}^{(1/2)}$	5.7250E-11
2.	PU, 0124d	20.7 (0.9)	$0.050 - 1.83E-4 \text{ time}^{(1/2)}$	7.5549E-11
3.	PTFEP, 153z	22.4 (0.9)	$0.083 - 2.92E-4 \text{ time}^{(1/2)}$	1.5448E-10
4.	PBB, 0138d	23.5 (1.0)	$0.056 - 1.91E-4 \text{ time}^{(1/2)}$	7.9684E-11
5.	PSB, 0140d	24.2 (1.0)	$0.042 - 1.44E-4 \text{ time}^{(1/2)}$	1.4751E-10
6.	PIP, 0131d	24.4 (1.0)	$0.070 - 2.38E-4 \text{ time}^{(1/2)}$	1.2030E-10
7.	PVC, 0089d	29.1 (1.2)	0.288 – 8.90E-4 time ^(1/2)	1.5727E-09
8.	SMO, 0129d	30.3 (1.3)	$0.209 - 6.33E-4 \text{ time}^{(1/2)}$	2.8522E-09
9.	PIB, 0137d	31.5 (1.3)	$0.041 - 1.21E-4 \text{ time}^{(1/2)}$	3.0844E-11
10.	PEEK, 154z	38.7 (1.6)	$0.163 - 4.38E-4 \text{ time}^{(1/2)}$	9.4139E-11
11.	PSBR, 143z	40.5 (1.7)	$0.081 - 2.11E-4 \text{ time}^{(1/2)}$	9.3694E-11
12.	ABS, 0115d	42.8 (1.8)	$0.124 - 3.15E-4 \text{ time}^{(1/2)}$	4.8703E-10
13.	PI, 0151z	46.4 (1.9)	$0.363 - 8.88E-4 \text{ time}^{(1/2)}$	1.1647E-10
14.	PMMA, 075z	53.8 (2.2)	$0.433 - 9.84E-4 \text{ time}^{(1/2)}$	1.8087E-09
15.	PC, 0117d	57.9 (2.4)	$0.119 - 2.60E-4 \text{ time}^{(1/2)}$	1.1518E-10
16.	PVC, 0110z	60.0 (2.5)	$0.175 - 3.77E-4 \text{ time}^{(1/2)}$	3.2042E-10
17.	PPO, 0091d	61.2 (2.5)	$0.151 - 3.22E-4 \text{ time}^{(1/2)}$	1.6204E-10
18.	PPO, 0096d	66.8 (2.8)	$0.092 - 1.88E-4 \text{ time}^{(1/2)}$	5.0719E-11
19.	PC, 0082d	72.2 (3.0)	$0.099 - 1.94E-4 \text{ time}^{(1/2)}$	5.7604E-11
20.	PDMS, 139d	83.0 (3.5)	$0.110 - 2.02E-4 \text{ time}^{(1/2)}$	1.1404E-10
21.	PMMA, 105z	130.1 (5.4)	$0.199 - 2.90E-4 \text{ time}^{(1/2)}$	1.6240E-10

Table 14. Number of Polymeric Materials in Various Cases Based on Desorption Relative to the Baseline and a Decrease or Plateau Approaching the Baseline

	Low Concentration	Plateau or Decreasing	Number of
Case	Desorption Relative To Zero	Desorption Approaching	Polymeric Materials
Code	Baseline	the Baseline	in Case, (% of Total)
NZP	<u>N</u> ear <u>Z</u> ero	Plateau	2 (9.5%)
NZD	Near Zero	Decreasing	1 (5%)
BZP	Below Zero	Plateau	3 (14%)
BZD	Below Zero	Decreasing	7 (33%)
AZP	Above Zero	Plateau	2 (9.5%)
AZD	Above Zero	Decreasing	6 (29%)

Most of the data reduction challenges from the diverse types of desorption plots were handled by the calculation procedures outlined in Table 9, with respect to assigning Case 1, 2, or 3 to the procedures. The NZP case in Table 14 is the simplest to analyze because the extrapolated time to complete desorption almost lies on the reduced mass plot. Likewise, the NZD case only requires a short extrapolation. Together, these only account for about 15% of the materials. About 47 % of the materials desorbed to below the zero mass baseline, indicate some extractables were present. These cases present a problem in defining the last mass data point for the reduced mass calculation. The BZP data sets can be handled by regressing that portion of the data set parallel to the baseline to define the onset of deviation from this plateau. This mass-time point would then be defined as the end of the desorption process. The low concentration diffusion would then be based on the Fickian regression of the last 3-10% of the data set above this reduced mass. The AZP cases are estimated to be caused by a shift in the specimen position and baseline. Again, this data case was handled by regressing that portion of the data set parallel to the baseline to define the onset of deviation from this plateau. The AZD case consists of materials with slow desorption for which the experiment was terminated early to increase the productivity of the instrument. If the data set did not extend to about 5% reduced mass remaining, the decontaminant-material might not have been in the low concentration Fickian region, and the extrapolation would lead to systematically low time-to-complete desorption. This case is best handled by repeating the experiment and establishing a criterion for terminating all experiments at less than about 5% mass remaining.

- Replicates. Measurement replicates and an analysis of reproducibility were not planned for this initial stage of the investigations; however, several paired specimens were evaluated to obtain some preliminary indications on trends or difficulties in data analysis. Four specimen pairs were evaluated: PVC, PMMA, PPO, and PC, and each is discussed below.
- <u>PVC</u>. The desorption time difference between the PVC repetitions is relatively high. Inspection of the Fick's Law and reduced mass versus time plots show a smooth, well-behaved approach to zero mass for both specimens. The PVC specimen with the longer desorption time had the higher initial carbon dioxide sorption (1.65 > 1.47%), and some part of the longer time might be attributed to this higher carbon dioxide content. The preferred PVC desorption time is the mean of 1.85 days.
- <u>PMMA</u>. A parallel comparison of the PMMA replicates yields a relatively high desorption time difference. Inspection of the low concentration Fick's Law diffusion extrapolation shows the lower desorption was produced by an extrapolation from about 20% from the baseline, whereas the longer complete desorption time extrapolation was from about 5%. Therefore, the 5.4 day "Time to Complete Desorption" is the preferred value (PMMA 105z). The longer desorption time was also from the specimen with the higher initial sorption value (6.4>5.8%).
- <u>PC</u>. The PC comparison of replicates shows a relatively small difference in initial sorption and desorption time. The Fick's Law plots overlay closely, even though there was balance instability at several time intervals (in PC 0082d). Because both replicates qualify as a statistical sample, the mean time of 2.7 days is the preferred value.

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• PPO. The PPO replicates belong to the AZP case; the curves came to a plateau slightly above the zero-mass baseline. The low concentration diffusion coefficient regression and extrapolation was heavily weighted by a large fraction of the data set with a significant, nominal positive mass. The "time to complete desorption" was, therefore, erroneously extended to longer time values. This case was handled by regressing the plateau portion of the data set parallel to the baseline to define the onset of deviation from this plateau. The onset of deviation was defined as the end of the desorption data set, and the low concentration regression was calculated from to onset to 3-10% higher residual mass values. The revised extrapolations are reported in Table 14. This data reduction procedure resulted in much closer agreement between replicates (2.5 versus 2.8 days). These data analysis procedures are being formalized for future studies but have not been auto-programmed into the data reduction software since some degree of judgment is still required.

### 3.5 Indentation Hardness.

Chemical exposure often influences the surface properties of materials that are critical to their performance. Indentation hardness is one of the most important of these surface properties. The values of the unexposed baseline measurements were compared with values after exposure and are documented in Table 15. About a third of the materials exposed to supercritical carbon dioxide showed surface hardening of about 1 to 3%. Another one-third experienced surface softening of over ca 1%. Note that the measurement shows the relatively low level of surface change after the plasticizing supercritical fluid had completely desorbed; measurements performed while a significant fraction of the decontaminant was still present resulted in larger changes. The methodology was capable of ranking the materials based on these longer-term indentation effects.

### 4. DISCUSSION

Sorption and desorption diffusion coefficient measurements by TGA were useful in characterizing the plasticization of polymeric materials by supercritical carbon dioxide. The sorption value measured includes solubility and void pressurization. The diffusion coefficient equations allowed a calculated estimate of the time required to return to the unplasticized state. The time determined to return to the initial unplasticized weight does not include the potential effects of residual trace carbon dioxide on glass transition temperature and crystallinity. Dynamic Mechanical Thermal Analysis (DMTA) studies of these effects as a function of desorption time are in progress. Indentation measurements showed slight changes after complete desorption. The continuous desorption curve measurement allowed adjustment and correction for extractables and the zero-time sorption measurement-lag after decompression; these corrections eliminated very high relative errors for many polymers.

The low concentration diffusion coefficient is sensitive to the interpretation of the plateau value near the zero baseline. The slope and diffusion coefficients have very low values of 10⁻⁹ to 10⁻¹⁰ cm²/sec for carbon dioxide. Therefore, the offset from the zero baseline due to extractables or slow equilibration will influence the extrapolations to a time-axis intercept. The goal is a set of data reduction procedures independent of operator interpretation. This process has been initiated by formulating several data analysis cases and rules, provided in Table 9 and 14.

Table 15. Indentation Hardness Changes to Polymeric Materials after Exposure to Supercritical Carbon Dioxide, Followed by Complete Desorption: Exposed to 1400 psig at 50 °C for 15 min

No.	Polymer Codes	Scale	Baseline	Final	Change, %
1.	PVC, 0110z	Shore D	ND	ND	ND
2.	PSB, 0140d	Shore D	63.6	45.8	-28.0
3.	PVC, 0089d	Shore D	73.4	71.5	-2.6
4.	PC, 0082d	Shore D	76.5	75.1	-1.8
5.	PMMA, 105z	Shore D	82.0	81.0	-1.2
6.	PC, 0117d	Shore D	76.9	76.1	-1.0
7.	PMMA, 075z	Shore D	80.2	79.5	-0.9
8.	PTFEP, 153z	Shore A	91.7	91.2	-0.5
9.	PIF, 0151z	Shore D	82.5	82.2	-0.4
10.	PU, 0124d	Shore A	96.9	97.0	0.1
11.	PEP, 0130d	Shore A	93.6	93.9	0.3
12.	PPO, 0096d	Shore D	75.6	75.8	0.3
13.	PPO, 0091d	Shore D	76.2	76.6	0.5
14.	SMO, 0129d	Shore A	58.6	58.9	0.5
15.	PEEK, 154z	Shore D	76.4	77.0	0.8
16.	ABS, 0115d	Shore D	72.6	73.4	1.1
17.	PSBR, 143z	Shore A	85.8	87.1	1.5
18.	PDMS, 139d	Shore A	63.9	64.9	1.6
19.	PIB, 0137d	Shore A	66.2	67.7	2.3
20.	PIP, 0131d	Shore A	66.9	68.7	2.7
21.	PBB, 0138d	Shore A	65.9	67.8	2.9

The sorption measurement desired is the value under supercritical conditions, however, the ability to measure values under these pressure and temperature conditions is currently limited. The relatively rapid 1-min decompression time maximizes our ability to measure the sorption as quickly as feasible. The ability to analyze polymer trends was confounded by attempting to use the sorption values without the corrections determined here. With these adjusted values, sorption trends can be correlated with structure, polymer solubility phase diagrams, or solvent-solute parameters. These correlations will assist in the selection of the optimal solvent class (supercritical or fluorocarbon) or temperature and pressure conditions. Classes or categories of materials that can or cannot withstand the cleaning process can also be identified. The further evaluation of the results can be provided in future publications.

There is an overall need for a measure of the desorption time required to deplasticize a material from sorbed decontaminant chemicals and return the material to its initial glass transition temperature and material properties that are dependent on plasticization and glass transition temperature. The diffusion measurements reported herein might be further exploited to provide a generalized limiting measure of the most rapid desorption feasible for any future decontaminant, because carbon dioxide is a favorable molecular prototype in terms of low

molecular cross-section. Therefore, most liquids such as fluorochemicals and solvent-based decontaminants (Decon Green) should have higher molecular cross-sections and slower returns to the initial glass transition temperature.

### 5. CONCLUSIONS

A methodology was developed and applied for evaluation of desorption diffusion of decontamination extractants such as supercritical fluids and fluorocarbons. The instrumentation and methods were capable of ranking polymeric materials based on interaction with supercritical carbon dioxide for several types of measurements. These measurements included: sorption, corrected for fraction of polymer additives extracted; sorption, corrected for extrapolation to the end of decompression; time for complete desorption to the initial weight; low-concentration diffusion coefficient; and change in surface indentation hardness.

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### LITERATURE CITED

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### APPENDIX A

### **COMPUTERIZED PROCEDURES**

Table Desorption Experiment Procedures for Thermogravimetric Instrumentation

Purpose	Steps
Initiate TA2100 Data acquisition	Reboot PC: Ctrl-Alt-Del
Initiate 1A2100 Data acquisition	Key F1 to select TGA mode
	Key F5 to Reconfigure, if needed
	ESC to enter Main Menu
Zero the amouty anasimon holder	Select F6: Signal Control
Zero the empty specimen holder	Select Fo. Signal Condo
WY ' 1 Al	
Weigh the specimen before chemical	Open Furnace, insert specimen, close furnace
exposure	Record weight on spreadsheet
	Remove specimen and submit to chemical
	exposure
Display and edit experimental parameters	Select F12 Instrument Control
	Select F4 Experimental Parameters
	Select Sample Info
	Select F1 Edit sample experimental parameters
	Scroll through and key in values
	Select F8 Accept Form
	ESC to Main Menu
Display and edit Method parameters	Select F2 Method
	Scroll to select Method
	ESC to finalize selection and return to Menu
Pre-equilibrate oven to desorption	Select F1 Start
temperature	
Pre-desorption experimental preparation	Key F12 Instrument Control
	Key Signal Control
	Key F1 Autozero
	Key F12 Instrument Control
	Key F3 Reject and Yes to end equilibration
Load chemically exposed specimen for	Weigh specimen on Macrobalance
desorption	Open furnace, load specimen, close furnace
	F4 to enter Elapsed time, Initial weights
	F1
•	Esc to Main Menu
	F1 Start
Display plot of data	Select F8 Utilities
	Select F1 Directory
	Select F3
	Esc
Update Data plot	F7 New Data Limit
	F1No Limit

Table Data Analysis File Transfer Procedures

	ysis File Transfer Procedures
Purpose	Steps
File transfer from TA2100 to DOS on same PC	• Reboot PC to exit TA2100: Alt-Ctrl-Del
Same PC	Insert diskette for data file
C. I. C. MARION	• F2 key to select DOS
Copy binary file from TA2100 directory	• Key "tadir data" DOS command to list TGA data files
to DOS directory	Note filename for transfer
	<ul><li>Key "taget filename" &gt;"1 File copied"</li></ul>
Convert DOS binary to ASCII format	• Key 'taconvrt", Enter; key in at prompt: "Name of
	binary TA data file:"
	• Key "filename.0#)" (pmma0123d.01)
	• ASCII: filename.01a (pmma0123d.01a)
Filter data get her calcuting at	Parameter Block: Key Yes
Filter data set by selecting the range and averaging set	• Data size: 1 (=All data)
averaging set	Data points: 3 (Least Square Average groups of N points)
	• Key point number (e.g. 50 for a plot, 3-9 for calculations)
	• Wait for prompt >
	Control-C to return to DOS Prompt
Transfer file to diskette	• Key "copy filename.01a a:"
Transfer file from diskette to PC with the	Highlight and Drag filename from a: diskette to
Diffusion Coefficient Data Analysis	DATA and ANALYSIS Folders
Program	
Convert data filename to code recognized	Open Analysis Folder, then open polymer.ini
by ANALIZ diffusion coefficient	• Create a New line at the end of the file, but before the
calculations	end-of-file control character.
	• Key in next Material Code by incrementing the current Code number
	Document the Material nomenclature employing
	conventions and delimiters
	• Click File, Save
Rename the TGA data file to the coded	• Open Analysis Folder
ANALIZ filename	• Highlight TGA filename,
	• Key in coded name
Convert TGA file to Coded ANALIZ file	Highlight Coded filename,
structure	Select Open in Word for large files
	• Delete first 20 lines of header parameters,
	• Delete any blank lines at top of data file, Delete any
	non-data lines at end of data file,
	• File>Save,
	• Select "Text document" (ASCII without control
	characters)
	• Close
	- 01000

Table Data Analysis File Transfer Procedures (Continued)

Purpose	Steps
Input specimen data for diffusion	Open INPUT database form;
calculations	• Key in coded filename,
	<ul> <li>Check that the correct polymer nomenclature is displayed.</li> </ul>
	<ul> <li>Key in specimen parameters as prompted,</li> </ul>
Set temporary value for initial weight at	<ul> <li>Key in the highest weight value from TGA plot or</li> </ul>
desorption zero-time	TGA data file
Complete the input portion of the database form	• Last input: "Time lapsed between sample blotting and loading" Or "Initial Durameter Hardness" when available;
	• File>Save>Close
Check for the creation of the correct file.dta filename in Analysis Folder	Open Analysis Folder or use Explorer>Find

Table Procedure for Calculation of Diffusion Coefficients

Table Procedure for Calculation of Diffusion Coefficients			
Purpose	Steps		
Calculate the extrapolated weight at zero-	Run ANALIZ		
time for desorption: Sample weight	• Key in filename (##-#-#)		
when removed from exposure fluid	• Examine 2 plots displayed		
	• Examine the data near the early data points for a linear		
	region to use for the extrapolation		
	• Examine the lower plot of the entire data set for		
	anomalies		
	• Key Enter/Any		
Define data range used for extrapolation	• Examine the Weight versus Square Root of Time plot		
	• Click on 2 points in the early desorption data; range		
	lines appear after the second click		
	• Key Enter		
Select the Extrapolated Weight at zero-	<ul> <li>Observe the display of regression parameters</li> </ul>		
time	<ul> <li>Determine if the y-intercept value is reasonable</li> </ul>		
	<ul> <li>Key in a weight at zero-time at the prompt</li> </ul>		
	• Key Enter, Control-C to exit		
Input the Extrapolated weight at zero-	Open INPUT		
time into INPUT database	Open filename ##-#-#		
	• Scroll to "Initial Weight of Specimen"		
	• Key in weight from y-intercept extrapolation		
	• File>Save		
Set up calculation of diffusion	Open ANALIZ		
coefficients using extrapolated weight at	Open filename		
zero-time	Key in Extrapolated weight again		

Table Procedure for Calculation of Diffusion Coefficients (Continued)

Purpose	Steps
Select the range of desorption data for calculation of the diffusion coefficient by Method 1	<ul> <li>Examine plot for linear Fickian region</li> <li>Key in "Yes" if there is a linear region for the calculation of a diffusion coefficient; for the Question Prompt: Is the plot Fickian?</li> <li>Key in values to define the calculation range in terms of fractional weight remaining</li> <li>Click on a high and low coordinates to define the range; repeating or altering the Keyed-in ranges</li> <li>Key in Enter</li> </ul>
Select ranges for Method 2, Low Concentration diffusion coefficient	<ul> <li>Examine the plot of the last 20% of the weight loss recorded; note, the relative weight might not include the complete curve</li> <li>Click on 2 coordinates on the plot to define the range</li> </ul>
Select ranges for a Series Solution for the diffusion coefficient	<ul> <li>Examine the plot of the natural log relative weight versus time</li> <li>Click on 2 points for calculation of an initial extrapolated weight</li> <li>Examine the replotted data set for selection of a linear range</li> <li>Click on 2 coordinates on the plot to select the range for the Series Solution calculation</li> </ul>
Record calculated results from the display	• Document value: "Time at 99% desorption flux", since this value is not printed from INPUT database
Document diffusion coefficient calculations	<ul> <li>Open INPUT database</li> <li>Select filename from File&gt;Open</li> <li>Print INPUT database form from File&gt;Print</li> <li>Exit</li> </ul>

## APPENDIX B

DESORPTION DIFFUSION COEFFICIENT DATA SETS AND COMPUTATIONS

**Supercritical Carbon Dioxide** and **Polycarbonate** 

## Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

04-1-1

TGA Filename:

pc0082d

PC

Material Name:

Property Test Specification: ASTM D471/D543

**Polycarbonate** 

Material Code:

PC

Material Supplier:

Harbor City Plastics

Material Class:

TP

Monomer Source:

General Electric

Base Polymer (Lot. No.): (LBR567)

Exposure Liquid:

Supercritical Carbon Dioxide

Replicate Number:

1

**Pre-Exposure Data** 

Measured Diameter (mm): Mean Diameter:

10.20

10.08

10.02

10.17 95% Confidence Level (CL):

0.131

<u>10.118</u> Measured Thickness (mils):

34.60 35.20

34.80

34.80

Mean Thickness:

34.850

79.822

0.400

Initial Sample Weight (mg): Initial Durometer Hardness:

76.5 Scale: SHORE D

95% Confidence Level (CL):

## Sample Exposure Data

**Exposure Date:** 

03/22/00

Time:

11:33:00

Removal Date: Total Exposure Time (h:m:s):

03/22/00

Time:

<u>11:48:00</u>

Weight of Sample After Exposure (mg): Percent Weight Gain:

81.70

Weight Gain (mg):

1.88

00:15:00

2.36

Final Durometer Hardness After Desorption:

75.1

Percent Hardness Change:

-1.8

#### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.000

95% CL:

0.0007

Time Lapse Between Sample Exposure and Sample Desorption (sec): Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

480

Sample Extracted (mg):

80.0

Solubility Corrected for Extractables (mg):

79.740 1.96

Sample Extracted (%):

0.10

Solubility Corrected for Extractables (%):

2.46

#### Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm²/sec):

1.6572E-07 1.00 - 0.65

Range Including Intercept (0,1), Relative Weight Fraction: Linear Range of Desorption, Relative Weight Fraction:

X-axis Intercept:

0.78 - 0.65

Slope: R Square:

R Square:

-1.0379E-02 0.99504

0.85851

Y-axis Intercept:

9.6722E+01 1.0038E+00

Low Concentration Diffusion Coefficient (cm²/sec):

5.7604E-11

Linear Range of Desorption, Relative Weight Fraction: Slope: -1.9350E-04

X-axis Intercept: Y-axis Intercept:

0.05 - 0.00 5.0965E+02 9.8616E-02

Wt. @ Zero	Wt. @ Zero	Adjustment to	Hardness
Time, mg	Time, %	TGA Time, sec	Shore Scale
K	L	M	, <b>N</b>
81.70	2.353	480	D
TGA Dry	Equil. Wt. Of	Min. Wt. Used	Pre Exposure
Wt., mg	Desorb. Sample	For FDC, mg.	Hardness
79.822	79.74	79.74	76.5
13.022	79.74	75.74	70.5
High Conc.	Low Conc.	<b>Total Liquid</b>	Post Desorption
DC, cm2/sec	DC, cm2/sec	Sorbed, mg.	Hardness
1.6572E-07	5.7604E-11	1.878	75.1
R Square	R Square		Change in
0.99504	0.85851		Hardness, %
			-1.8
X Intercept	X Intercept		
96.72	509.6		Mean Thickness,
V lataura and	V (-1		cm.
Y Intercept	Y Intercept		0.0885
1.00383	0.09862		
W to be seen a made	V (-1	V 1-1	pc0082d
Y Intercept	X Intercept	X Intercept	X Intercept
as Wt., mg.	as Time, hrs.	as Time, min.	as Time, days
82.013	72.2	432.9	3.0
		Max. TGA Wt.,	TGA Wt. At 20
TEMPERATURE STATIST	TICS	(orig data file)	min. Adjustment
		81.296	81.004
Column1			
		Sorption @ 7-12	Sorption @ 20
Mean	29.9997751	min., mg	min., mg
Standard Error	0.000349877	1.47	1.18
Median	30	0 " 0 = 40	
Mode	30.001	Sorption @ 7-12	Sorption @ 20
Standard Deviation	0.022304498	min., %	min., %
Sample Variance	0.000497491	1.85	1.48
Kurtosis	1237.843422	Dal Funa (9/) of	Dal Eman (0/) of
Skewness		Rel. Error (%) of	Rel. Error (%) of
Range	1.222	7-12 min. Sorp.	20 min. Sorp
Minimum	28.983	-21.5	-37.1
Maximum	30.205		
Sum	121919.086		
Count	4064		
Confidence Level(95.0%)	0.000685951		

## HIGH CONCENTRATION DIFFUSION COEFFICIENT

pc0082d

### **SUMMARY OUTPUT**

Regression Statistics				
Multiple R	0.997515832			
R Square	0.995037835			
Adjusted R Square	0.994822089			
Standard Error	0.005089135			
Observations 25				

## **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.119449449	0.119449449	4612.07371	5.21878E-28
Residual	23	0.000595684	2.58993E-05		
Total	24	0.120045133			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	1.00383392	0.004326717	232.0082228	2.935E-40	0.994883437	1.012784404
X Variable 1	-0.010378585	0.000152823	-67.91225005	5.2188E-28	-0.010694724	-0.010062446

# LOW CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

Regression Statistics				
Multiple R	0.92655813			
R Square	0.858509968			
Adjusted R Square	0.858383975			
Standard Error	0.005271814			
Observations	1125			

## ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.18937359	0.18937359	6813.95486	0
Residual	1123	0.031210442	2.7792E-05		
Total	1124	0.220584032			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.098615531	0.001046021	94.27682236	0	0.096563154	0.100667908
X Variable 1	-0.000193497	2.34409E-06	-82.54668293	0	-0.000198096	-0.000188898

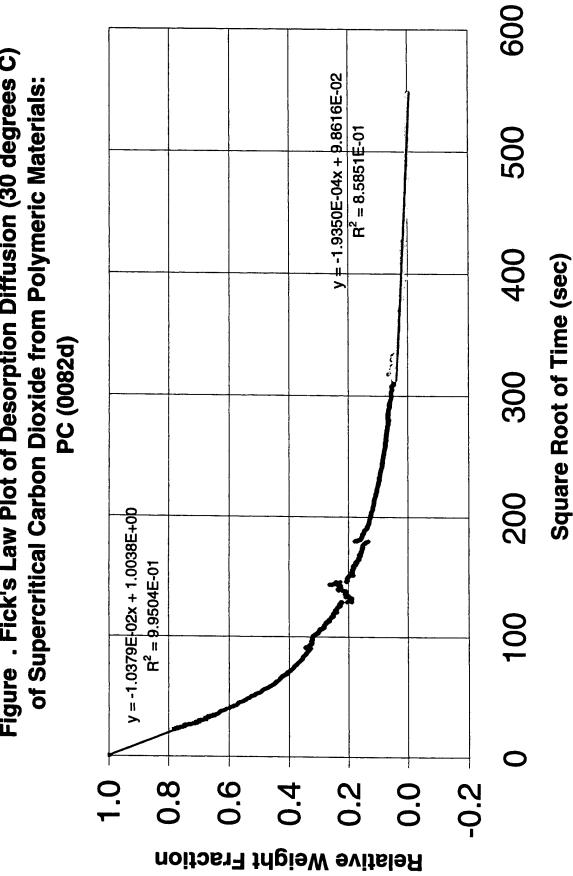
Column1			
Mean	10.1175		
Standard Error	0.04130678		
Median	10.125		
Mode	#N/A		
Standard Deviation	0.08261356		
Sample Variance	0.006825		
Kurtosis	-3.032967		
Skewness	-0.3232314		
Range	0.18		
Minimum	10.02		
Maximum	10.2		
Sum	40.47		
Count	4		
Confidence Level(95.0%)	0.13145673		

## THICKNESS STATISTICS

	Column1	Column1		
	Mean	34.85		
	Standard Error	0.12583057		
	Median	34.8		
	Mode	34.8		
	Standard Deviation	0.25166115		
	Sample Variance	0.06333333		
	Kurtosis	2.22714681		
(LBR567)	Skewness	1.12933811		
pc0082d	Range	0.6		
•	Minimum	34.6		
	Maximum	35.2		
	Sum	139.4		
	Count	4		
	Confidence Level(95.0%)	0.40044942		

ND/value ND/value A/D

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:





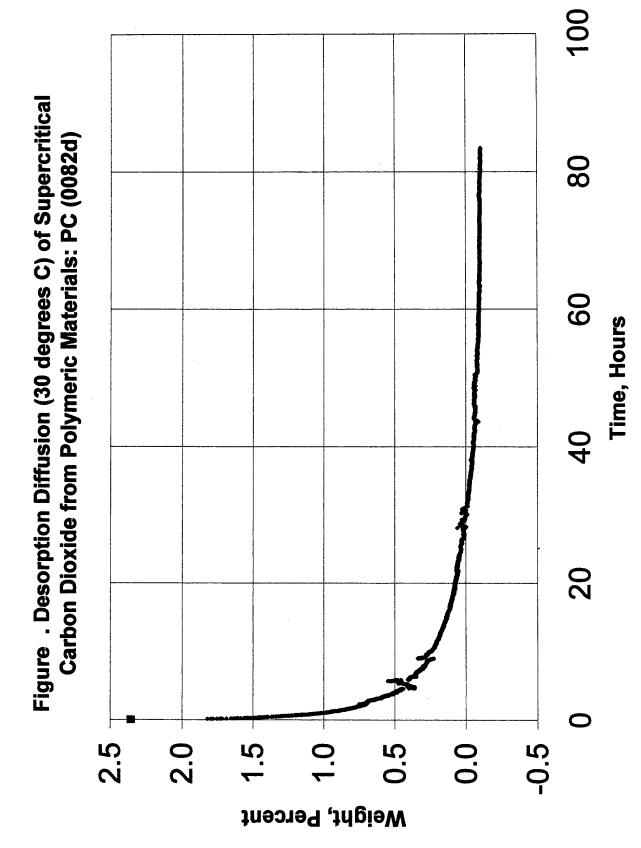
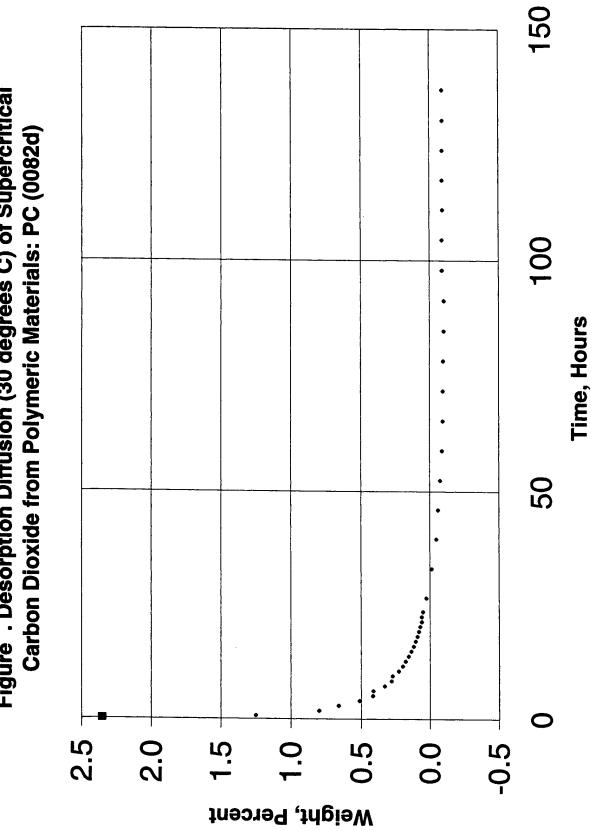
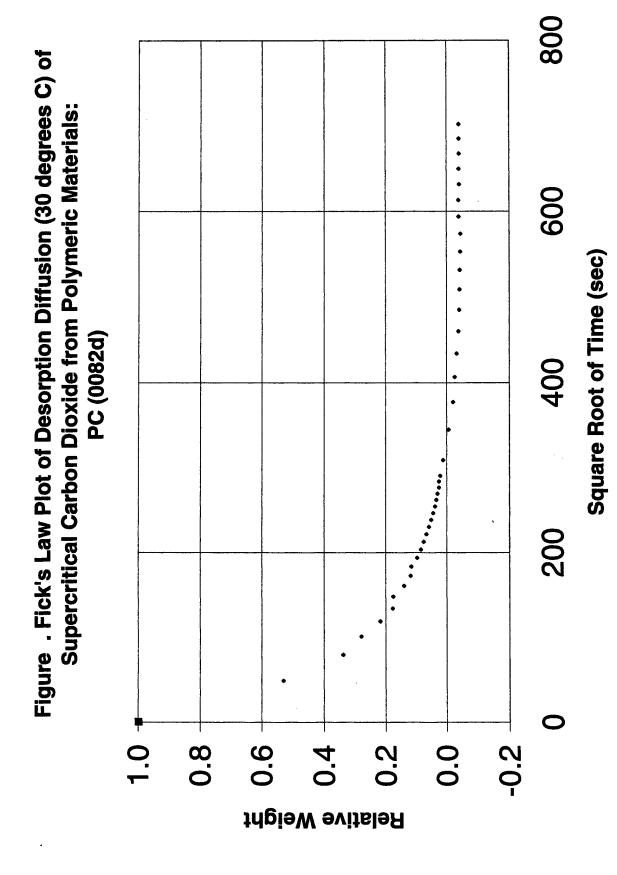


Figure . Desorption Diffusion (30 degrees C) of Supercritical





Hardness	Scale	2 10	0 800	Pre Exposure	Hardness	76.5		Post Desorption	Hardness	75.1		Change in	Hardness, %	-1.83															2 ET: 7:45												
Adjustment to	TGA Time, sec	₹ 6	ě				4																						Comment Disk: .4x0.03 in. HP@1400x50 Dry Wt, TGA: 79.822 ET: 7:45												
TGA Dry	Wt., mg	10 CC	19.026		f Data	sumi	ginning with									_			:0082d.01	¥							mal		@ 1400x50 E												
Wt. @ Zero	Time, %	2 0	7.333		Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4			€	Q Q	<u>(</u> :	Hn = (((Cn - L4) / L4) * 100)	/ (J4 - L4))	1) / L4) * 100)	N16 = (((N12 - N8) / N8) * 100)			Source binary TA data file: pc0082d.01	14 lines in the parameter block	f data			Module TGA Aluminum Pans	CO2 0082D	Ō	Method DESORPTION-Isothernal		c: .4x0.03 in. HP		2	ture (øC)	ng)		0						
>	Time, mg	· •	07:10		Formulae Use	Uppercase let	Lowercase n		Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L	$\ln = ((Cn - L4) / (J4 - L4))$	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12.			Source binary	14 lines in the	3 channels of data		Run 22	Module TGA A	Sample PC SCCO2 0082D	Size 81.296 mg	Method DESO	Operator WJS	Comment Disk	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 22-Mar-00	Time 11:56					
	Wt. Loss	- 4	0.339	0.280	0.218	0.177	0.176	0.141	0.119	0.117	0.097	0.086	0.076	0.067	0.029	0.051	0.045	0.039	0.034	0.031	0.026	0.026	0.022	0.012	9000	-0.021		_						-0.045	_	-0.039	-0.041	-0.040	0.040	-0.040	-0.040
Wt. Loss,	<b>%</b> =	- <del>-</del> -	0.797	0.659	0.512	0.417	0.413	0.331	0.281	0.275	0.229	0.201	0.179	0.157	0.138	0.121	0.106	0.092	0.081	0.072	0.061	0.061	0.051	0.028	-0.015	-0.049	-0.061	-0.077	-0.091	960.0	-0.098	6. 100	-0.105	<del>.</del> 0.106	-0.092	-0.092	-0.096	-0.094	-0.094	-0.095	-0.094
SQRT Adj. Wt. Loss,	Time, sec	5 6	79.55	101.17	118.91	134.33	148.14	160.78	172.49	183.47	193.82	203.64	213.01	222.00	230.62	238.93	246.97	254.75	262.30	269.64	276.79	283.76	290.58	308.85	344.35	376.80	406.67	434.49	460.63	485.37	508.90	531.40	552.97	573.74	593.79	613.17	631.97	650.22	667.97	685.26	702.13
Adjusted	Time, hr	- 60	1.758	2.843	3.928	5.013	8.096	7.181	8.265	9.350	10.435	11.519	12.604	13.690	14.774	15.858	16.942	18.027	19.112	20.196	21.282	22.367	23.451	26.497	32.938	39.438	45.938	52.438	58.938	65.439	71.939	78.439	84.939	91.439	97,939	104.439	110.940	117.440	123.940	130.440	136.941
Adjusted	Time, sec	74.0	6327.840	10235.460	14139.060	18045.180	21946.800	25849.980	29754.240	33659.820	37564.500	41469.840	45375.240	49282.200	53185.560	57088.680	60992.940	64898.220	68801.760	72706.860	76614.060	80522.100	84425.160	95390.940	118575.960	141976.080	165377.700	188777.580	212177.460	235579.860	258980.160	282380.760	305780.940	329181.900	352581.480	375981.180	399383.820	422784.360	446184.840	469585.080	492986.040
TGA Time,	S9 C	104	5847.840	9755.460	13659.060	17565.180	21466.800	25369.980	29274.240	33179.820	37084.500	40989.840	44895.240	48802.200	52705.560	56608.680	60512.940	64418.220	68321.760	72226.860	76134.060	80042.100	83945.160	94910.940	118095.960	141496.080	164897.700	188297.580	211697.460	235099.860	258500.160	281900.760	305300.940	328701.900	352101,480	375501,180	398903.820	422304.360			492506.040
TGA	Wt. mg	9	80.458	80.348	80.231	80.155	80.152	80.086	80.046	80.041	80.005	79.983	79.965	79.948	79.932	79.919	79.907	79.896	79.887	79.880	79.871	79.871	79.863	79.844	79.810	79.783	79.773	79.761	79.750	79.745	79.744	79.742	79.738	79.738	79.749	79.749	79.745	79.747	79.747	79.746	79.747
Temp.	O geb	200	30.000	29.999	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.00	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000
TGA Time,	Ē <	200	97.464	162.591	227.651	292.753	357.780	422.833	487.904	552.997	618.075	683.164	748.254	813.370	878.426	943.478	1008.549	1073.637	1138.696	1203.781	1268.901	1334.035	1399.086	1581.849	1968.266	2358.268	2748.295	3138.293	3528.291	3918.331	4308.336	4698.346	5088.349	5478.365	5868.358	6258.353	6648.397	7038.406	7428.414	7818.418	8208.434

**Supercritical Carbon Dioxide** 

and

Polycarbonate

PC

#### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

04-1-2

TGA Filename:

pc0117d

Property Test Specification: ASTM D471/D543 Material Name:

Polycarbonate

Material Code:

Material Supplier:

**Harbor City Plastics** 

Material Class:

Monomer Source:

General Electric

Base Polymer (Lot. No.): (LBR567)

Exposure Liquid:

Replicate Number:

2

Supercritical Carbon Dioxide

**Pre-Exposure Data** 

Measured Diameter (mm):

10.26

10.16

10.08 10.22 95% Confidence Level (CL):

0.125

Mean Diameter: Measured Thickness (mils):

10.190

36.60 37.60 36.20

36.10

Mean Thickness:

36.625

00:15:00

95% Confidence Level (CL):

1.090

Initial Sample Weight (mg): Initial Durometer Hardness: <u>83.130</u> 76.9

Scale: SHORE

D

#### **Sample Exposure Data**

**Exposure Date:** 

04/26/00

Time:

14:32:00

Removal Date:

04/26/00

Time:

14:47:00 Weight of Sample After Exposure (mg):

85.20

Total Exposure Time (h:m:s):

2.07

Percent Weight Gain:

2.49

Weight Gain (mg):

Percent Hardness Change:

Final Durometer Hardness After Desorption:

<u>76.1</u>

-1.0

### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.000

95% CL:

0.0006

Time Lapse Between Sample Exposure and Sample Desorption (sec): Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

505

Sample Extracted (mg):

0.11

Solubility Corrected for Extractables (mg):

83.020

Sample Extracted (%):

0.13

Solubility Corrected for Extractables (%):

2.18 2.62

#### Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm2/sec):

Range Including 0,1 Intercept, Relative Weight Fraction:

2.1310E-07 1.00 - 0.62

Slope:

-1.1199E-02

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.75 - 0.62

R Square:

0.99947

Y-axis Intercept:

8.9294E+01 9.9998E-01

Low Concentration Diffusion Coefficient (cm2/sec):

1.1518E-10

Slope:

-2.6035E-04

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.05 - 0.00

R Square:

0.95361

Y-axis Intercept:

4.5669E+02 1.1890E-01

Wt. @ Zero Time, mg K 85.20	Wt. @ Zero Time, % L 2.490	Adjustment to TGA Time, sec M 505	Hardness Shore Scale N D
TGA Dry Wt., mg 83.130	Equil. Wt. Of Desorb. Sample 83.02	Min. Wt. Used For FDC, mg. 83.020	Pre Exposure Hardness 76.9
High Conc. DC, cm2/sec 2.1310E-07	Low Conc. DC, cm2/sec 1.1518E-10	Total Liquid Sorbed, mg. 2.070	Post Desorption Hardness 76.1
R Square 0.99947 X Intercept	R Square 0.95361		Change in Hardness, % -1.0
89.29	X Intercept 456.7		Mean Thickness, cm.
Y Intercept 0.9998	Y Intercept 0.11890		0.0930
Y Intercept as Wt., mg. 85.198	X Intercept as Time, hrs. 57.9	X Intercept as Time, min. 347.6	pc0117d X Intercept as Time, days 2.4
TEMPERATURE STATIS		Max. TGA Wt., (orig data file) 84.665	TGA Wt. At 20 min. Adjustment 84.362
Column1			_
Mean Standard Error Median	30.00019721 0.000308961 30	Sorption @ 7-12 min., mg 1.54	Sorption @ 20 min., mg 1.23
Mode Standard Deviation		Sorption @ 7-12 min., %	Sorption @ 20 min., %
Sample Variance Kurtosis	0.000356244 885.3566777	1.85	1.48
Skewness Range	-24.1727843 0.952	7-12 min. Sorp.	Rel. Error (%) of 20 min. Sorp
Minimum Maximum Sum	29.244 30.196	-25.8	-40.5
Count Confidence Level(95.0%)	111960.736 3732 0.000605747		

## HIGH CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.999735807								
R Square	0.999471684								
Adjusted R Square	0.999447669								
Standard Error	0.001800319								
Observations	24								

#### ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.134895688	0.134895688	41619.7136	1.50569E-37
Residual	22	7.13053E-05	3.24115E-06		
Total	23	0.134966994			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.999979835	0.001562048	640.1724278	1.7934E-48	0.996740343	1.003219327
X Variable 1	-0.011198696	5.48931E-05	-204.0091018	1.5057E-37	-0.011312537	-0.011084854

### LOW CONCENTRATION DIFFUSION COEFFICIENT

### **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.976527763								
R Square	0.953606472								
Adjusted R Square	0.953561254								
Standard Error	0.003773243								
Observations	1028								

# <u>ANOVA</u>

	df	SS	MS	F	Significance F
Regression	1	0.300253906	0.300253906	21089.1536	0
Residual	1026	0.014607533	1.42374E-05		
Total	1027	0.314861439			

<u> </u>	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.118901214	0.000694648	171.1674609	0	0.117538119	0.120264309
X Variable 1	-0.000260352	1.7928E-06	-145.221051	0	-0.00026387	-0.000256834

Column1	
Mean	10.18
Standard Error	0.0391578
Median	10.19
Mode	#N/A
Standard Deviation	0.0783156
Sample Variance	0.00613333
Kurtosis	-0.768431
Skewness	-0.5995807
Range	0.18
Minimum	10.08
Maximum	10.26
Sum	40.72
Count	4
Confidence Level(95.0%)	0.12461771

ND/value ND/value A/D

# THICKNESS STATISTICS

	Column1	
	Mean	36.625
	Standard Error	0.34247871
	Median	36.4
	Mode	#N/A
	Standard Deviation	0.68495742
	Sample Variance	0.46916667
	Kurtosis	1.82944073
(LBR567)	Skewness	1.46331586
pc0117d	Range	1.5
	Minimum	36.1
	Maximum	37.6
	Sum	146.5
	Count	4
	Confidence Level(95.0%)	1.08992113

APPENDIX B

APPENDIX B

51

Relative Weight Fraction

900 Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: 500 y = -2.60354 - 04x + 1.1890E - 01 $R^2 = 9.5361E-01$ 400 PC (0117d) 300 200 y = -1.1199E - 02x + 9.9998E - 01 $R^2 = 9.9947E-01$ 100 0.6 -0.2 0.8 0.2 0.4 0.0

Square Root of Time (sec)

Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PC (0117d)

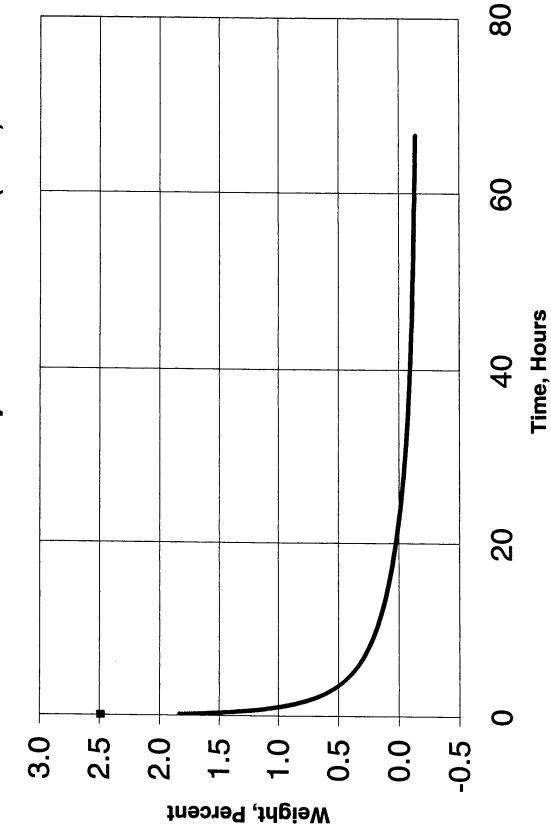
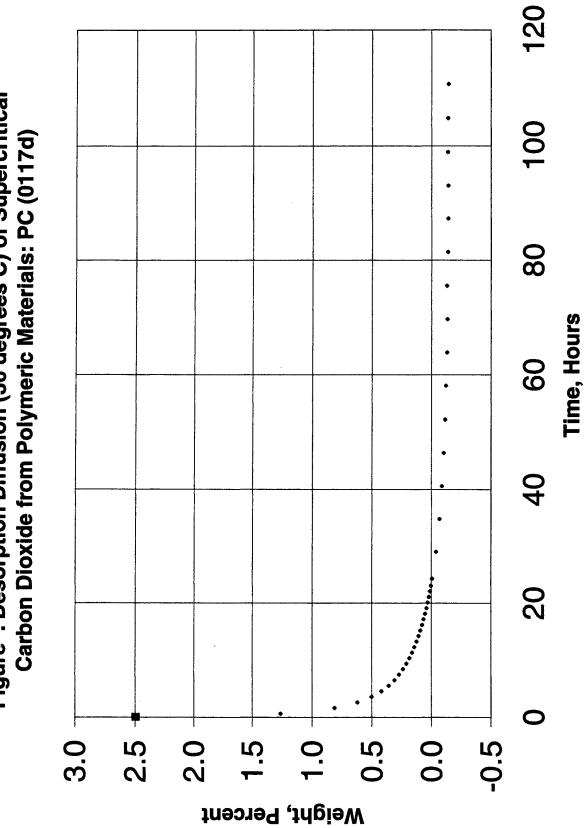
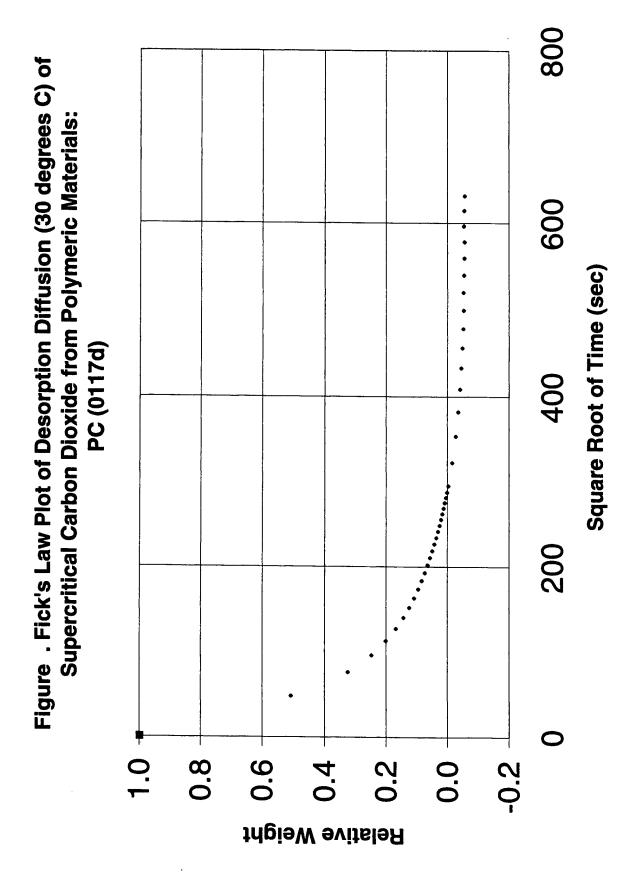


Figure . Desorption Diffusion (30 degrees C) of Supercritical





Hardness	S S	Shore D		Pre Exposure	Hardness	76.9		Post Desorption	Hardness	76.1		Change in	Hardness, %	49.															I-82.96 ET: 8:15												
Adjustment to	M (5)	505					h n=4	•																					Comment Disk: .4x0.03in., HP@1400x50, Dry: TG-83.125 M-82.96 ET: 8:15												
TGA Dry	5	83.125			of Data	olumns	beginning wil									6			pc0117d.01	ock		1		ç,			hermal		1P@1400x5												
Wt. @ Zero	, , ,	2.496			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4		_	æ	Q.	.i.	⊣n = (((Cn - L4) / L4) * 100)	/ (J4 - L4))	1)/ L4) * 100)	- N8) /N8) * 1			TA data file:	14 lines in the parameter block	f data	***************************************		<b>Vuminum Par</b>	CC02 0117D	5	RPTION-Isot		c: .4x0.03in., }		ĉ	ture (øC)	ng)		0						
Wt. @ Zero	) () ()	85.20			Formulae Use	Uppercase let	Lowercase n r	***************************************	Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L	$\ln = ((Cn - L4) / (J4 - L4))$	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8)/N8) * 100)		٠	Source binary TA data file: pc0117d.01	14 lines in the	3 channels of data		Run 33	Module TGA Aluminum Pans	Sample PC SCCO2 0117D	Size 84.665 mg	Method DESORPTION-Isothermal	Operator BSI	Comment Disk	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	<b>Jate 26-Apr-00</b>	Firne 14:56					
Fractional Wt Loss	-	0.507	0.325	0.248	0.201	0.169	0.144	0.125	0.109	960.0	0.085	0.074	0.065	0.057	0.050	0.043	0.037	0.031	0.026	0.021	0.017	0.013	0.009	0.005	0.001	-0.003	_	_		_				-0.053	-0.052	-0.055	-0.056	-0.056	-0.054	-0.055	-0.057
Wt. Loss,	? I	1.265	0.812	0.619	0.502	0.421	0.360	0.312	0.273	0.240	0.211	0.186	0.163	0.143	0.125	0.108	0.093	0.079	0.065	0.053	0.042	0.031	0.022	0.012	0.003	-0.007	-0.039	-0.068	-0.088	-0.102	-0.112	-0.121	-0.129	-0.132	-0.129	-0.136	-0.139	-0.139	-0.135	-0.138	-0.142
SORT Adj. Wt. Loss,	) (0	47.43	75.83	96.19	112.95	127.52	140.59	152.54	163.62	174.00	183.79	193.09	201.97	210.46	218.63	226.50	234.11	241.47	248.63	255.58	262.34	268.93	275.36	281.65	287.80	295.32	322.70	353.75	382.28	408.83	433.75	457.32	479.73	501.15	521.68	541.43	560.49	578.92	596.78	614.13	630.99
Adjusted Time hr	Ē	0.625	1.597	2.570	3.544	4.517	5.490	6.463	7.436	8.410	9.383	10.357	11.331	12.304	13.277	14.251	15.224	16.197	17.171	18.144	19.117	20.090	21.062	22.035	23.008	24.226	28.927	34.761	40.594	46.428	52.262	58.096	63.929	. 69.763	75.596	81.430	87.263	93.097	98.931	104.765	110.598
Adjusted Time sec	Э ш	2249.980	5750.560	9252.460	12757.480	16262.200	19765.360	23268.340	26770.360	30274.660	33778.840	37285.120	40789.960	44293.180	47798.380	51304.000	54806.320	58308.880	61814.620	65318.860	68821.060	72322.900	75824.020	79327.780	82829.860	87215.140	104136.940	125138.560	146139.040	167140.000	188142.160	209143.840	230144.560	251146.360	272146.900	293147.500	314147.680	335149.000	356151.580	377153.200	398154.520
TGA Time,	<u> </u>	1744.980	5245.560	8747.460	12252.480	15757.200	19260.360	22763.340	26265.360	29769.660	33273.840	36780.120	40284.960	43788.180	47293.380	50799.000	54301.320	57803.880	61309.620	64813.860	68316.060	71817.900	75319.020	78822.780	82324.860	86710.140	103631.940	124633.560	145634.040	166635.000	187637.160	208638.840	229639.560	250641.360	271641.900	292642.500	313642.680	334644.000	355646.580	376648.200	397649.520
TGA W	ပ	84.177	83.800	83.640	83.543	83.475	83.424	83.384	83.352	83.324	83.301	83.279	83.261	83.244	83.229	83.215	83.202	83.190	83.179	83.169	83.160	83.151	83.143	83.135	83.128	83.119	83.093	83.069	83.052	83.040	83.032	83.024	83.018	83.016	83.018	83.012	83.010	83.009	83.013	83.011	83.007
Temp.	) (2)	29.992	30.001	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.005	30.005	30.005	30.000	30.000	30.003	30.001	30.000	30.000	30.003	30.002
TGA Time,	∢	29.083	87.426	145.791	204.208	262.620	321.006	379.389	437.756	496.161	554.564	613.002	671.416	729.803	788.223	846.650	905.022	963.398	1021.827	1080.231	1138.601	1196.965	1255.317	1313.713	1372.081	1445.169	1727.199	2077.226	2427.234	2777.250	3127.286	3477.314	3827.326	4177.356	4527.365	4877.375	5227.378	5577.400	5927.443	6277.470	6627.492

Blank

**Supercritical Carbon Dioxide** 

and

**Polymethyl Methacrylate** 

**PMMA** 

## Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

01-1-2

TGA Filename:

pmma105z

Material Name:

Property Test Specification: ASTM D471/D543 Polymethyl Methacrylate

Material Code:

**PMMA** 

Material Supplier:

**Harbor City Plastics** 

**Material Class:** 

TP

Monomer Source:

Rohm & Haas

Base Polymer (Lot. No.): Plexiglass (76B818)

Exposure Liquid:

Supercritical Carbon Dioxide

Replicate Number:

**Pre-Exposure Data** 

Measured Diameter (mm): Mean Diameter:

10.41

10.42

10.43 95% Confidence Level (CL):

0.021

Measured Thickness (mils):

38.30

39.40 39.40

39.00

Mean Thickness:

39.025

10.425

91.020

00:15:00

95% Confidence Level (CL):

10.44

0.826

Initial Sample Weight (mg): Initial Durometer Hardness:

82.0

Scale: SHORE

D

#### Sample Exposure Data

**Exposure Date:** 

04/14/00

Time:

09:50:00

Removal Date:

04/14/00

Time:

10:05:00

Total Exposure Time (h:m:s):

Weight of Sample After Exposure (mg):

96.70

Weight Gain (mg):

5.68

Percent Weight Gain:

6.24

Final Durometer Hardness After Desorption:

81.0

Percent Hardness Change:

-1.2

#### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.000

95% CL:

0.0006

Time Lapse Between Sample Exposure and Sample Desorption (sec): Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

<u>480</u>

Sample Extracted (mg):

-0.20

Solubility Corrected for Extractables (mg):

91.220

Sample Extracted (%):

-0.22

Solubility Corrected for Extractables (%):

5.68 6.24

## **Diffusion Coefficient Data: Fick's Law**

High Concentration Diffusion Coefficient (cm²/sec):

9.6726E-08 1.00 - 0.70

Linear Range of Desorption, Relative Weight Fraction:

-7.0808E-03

X-axis Intercept:

0.84 - 0.70

Slope: R Square:

0.99879

Y-axis Intercept:

1.4056E+02 9.9531E-01

Low Concentration Diffusion Coefficient (cm²/sec):

Range Including Intercept (0,1), Relative Weight Fraction:

1.6240E-10

Slope:

-2.9014E-04

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.07 - 0.046.8436E+02

R Square: 0.98778

Y-axis Intercept:

1.9856E-01

Wt. @ Zero	Wt. @ Zero	Adjustment to	Hardness
Time, mg	Time, %	TGA Time, sec	Shore Scale
K 96.70	L 6.040	M	N
90.70	6.240	480	D
TGA Dry	Equil. Wt. Of	Min. Wt. Used	Pre Exposure
Wt., mg	Desorb. Sample	For FDC, mg.	Hardness
91.020	91.22	91.020	82.0
High Conc.	Low Conc.	Total Liquid	Post Desorption
DC, cm2/sec	DC, cm2/sec	Sorbed, mg.	Hardness
9.6726E-08	1.6240E-10	5.680	81.0
R Square	R Square		Ohaman in
0.99879	0.98778		Change in
0.00070	0.90776		Hardness, % -1.2
X Intercept	X Intercept		1.6
140.56	684.4		Mean Thickness,
	33		cm.
Y Intercept	Y Intercept		0.0991
0.99531	0.19856		
V (-1			pmma105z
Y Intercept	X Intercept	X Intercept	X Intercept
as Wt., mg. 96.246	as Time, hrs.	as Time, min.	as Time, days
30.240	130.1	780.6	5.4
		Max. TGA Wt.,	TGA Wt. At 20
TEMPERATURE STATIS	TICS	(orig data file)	min. Adjustment
		95.811	95.271
Column1		Sorption @ 7-12	Cambia @ 00
Mean	30.00010643	min., mg	Sorption @ 20
Standard Error	0.000290767	4.79	min., mg 4.25
Median	30	4.70	4.25
Mode	29.999	Sorption @ 7-12	Sorption @ 20
Standard Deviation	0.018843886	min., %	min., %
Sample Variance	0.000355092	5.26	4.67
Kurtosis	633.726776	0.20	4.07
Skewness	-15.92093413	Rel. Error (%) of	Rel. Error (%) of
Range	0.948	7-12 min. Sorp.	20 min. Sorp
Minimum	29.304	-15.7	-25.2
Maximum	30.252	10.7	- <u>2</u> 0,2
Sum	126000.447		
Count	4200		
Confidence Level(95.0%)	0.000570058		

## pmma105z

## HIGH CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

Regression Statistics										
Multiple R	0.999392912									
R Square	0.998786192									
Adjusted R Square	0.998757963									
Standard Error	0.001910724									
Observations	45									

## ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.129177474	0.129177474	35382.6889	2.46696E-64
Residual	43	0.000156987	3.65087E-06		
Total	44	0.129334462			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.995306197	0.001254388	793.4594157	3.3271E-91	0.992776484	0.99783591
X Variable 1	-0.007080764	3.7643E-05	-188.1028679	2.467E-64	-0.007156678	-0.00700485

## LOW CONCENTRATION DIFFUSION COEFFICIENT

## SUMMARY OUTPUT

Regression Statistics									
Multiple R	0.993869749								
R Square	0.987777077								
Adjusted R Square	0.987758669								
Standard Error	0.001092044								
Observations	666								

## **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.063992957	0.063992957	53660.1588	0
Residual	664	0.00079186	1.19256E-06		
Total	665	0.064784817			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.198557182	0.000644294	308.1780092	0	0.197292082	0.199822282
X Variable 1	-0.000290135	1.25249E-06	-231.6466247	0	-0.000292594	-0.000287676

Column1	
Mean	10.425
Standard Error	0.00645497
Median	10.425
Mode	#N/A
Standard Deviation	0.01290994
Sample Variance	0.00016667
Kurtosis	-1.2
Skewness	4.1231E-13
Range	0.03
Minimum	10.41
Maximum	10.44
Sum	41.7
Count	4
Confidence Level(95.0%)	0.02054262

## THICKNESS STATISTICS

	Column1	
	Columni	
	Mean	39.025
	Standard Error	0.25940637
	Median	39.2
	Mode	39.4
	Standard Deviation	0.51881275
	Sample Variance	0.26916667
	Kurtosis	1.030931
Plexiglass (76B818)	Skewness	-1.3158168
pmma105z	Range	1.1
	Minimum	38.3
	Maximum	39.4
	Sum	156.1
	Count	4
	Confidence Level(95.0%)	0.82554763

ND/value ND/value A/D

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: **PMMA (105z)** 

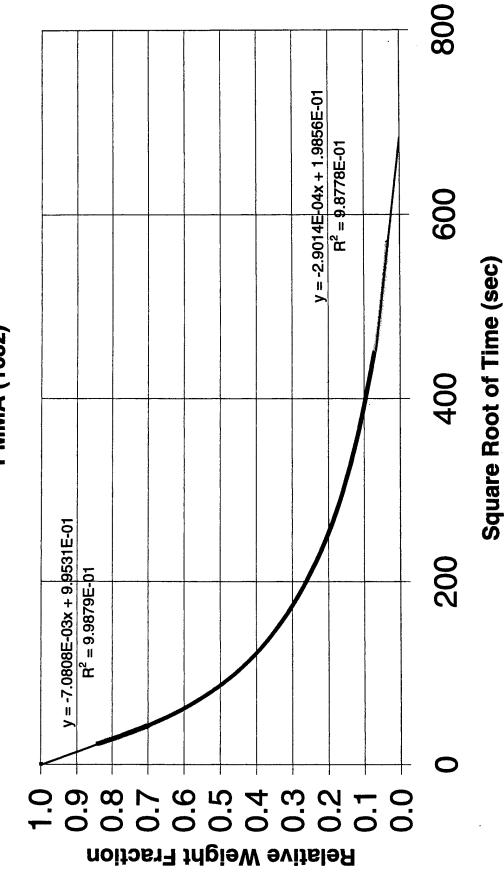


Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PMMA (105z)

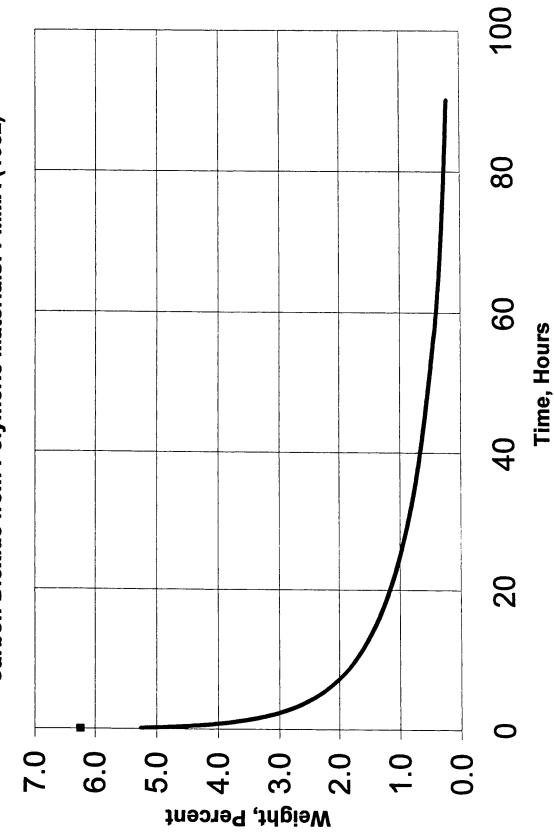


Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PMMA (105z)

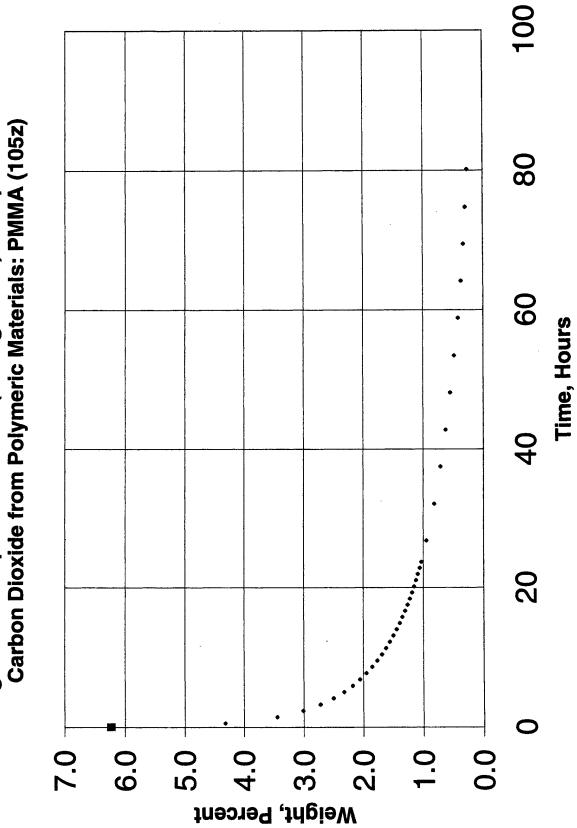
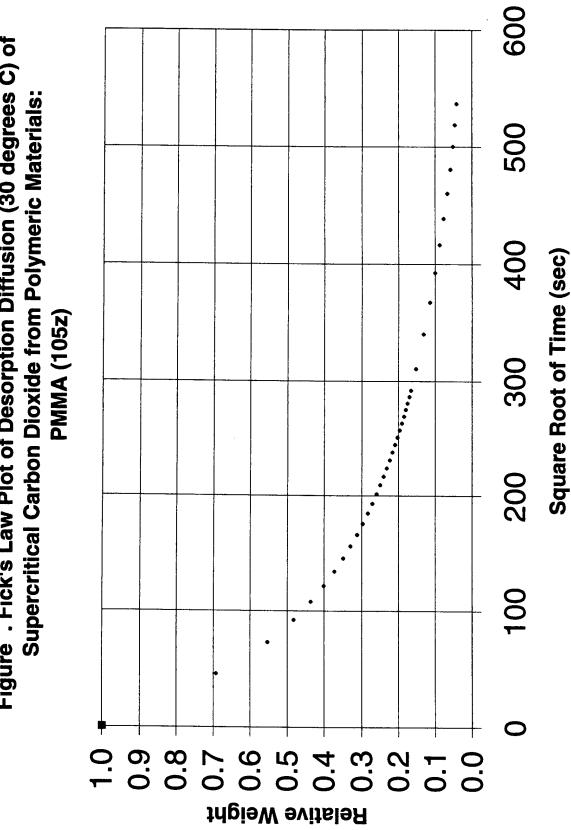


Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



Hardness	2	Shore D		Pre Exposure	Hardness	82.0		Post Desorption	Hardness	81.0		Change in	Hardness, %	-1.22															VI: 91.20 ET:8.00										
Adjustment to	<b>™</b>	480					n=4																						Comment Disk: .4x0.03 in. HP@1400x50, DRY: TG: 91.022, M: 91.20 ET:8.00										
TGA Dry	6	91.022			of Data	lumns	eginning with									Q Q			mma105z.01	SCK SCK				<b>"</b>	1105Z		ermal		IP@1400x50,										
Wt. @ Zero	, , ,	6.238			I for Analysis	ers refer to co	fers to row, b	**************			6	Ē	() / L4) * 100)	/ (J4 - L4))	/ L4) * 100)	N8) /N8) * 10			TA data file : p	parameter blo	data			fuminum Pans	SCO2 PMMA	m	RPTION-Isoth		: .4x0.03 in. H		<del>-</del>	ure (øC)	ıg)		0				
Wt. @ Zero	) 	96.70			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4	**************	Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	n = ((Cn - L4) / (J4 - L4))	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8) /N8) * 100)			Source binary TA data file: pmma105z.01	14 lines in the parameter block	3 channels of data		Run 28	Module TGA Aluminum Pans	Sample PMMA SCO2 PMMA105Z	Size 95.811 mg	Method DESORPTION-Isothermal	Operator WJS	Comment Disk	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 14-Apr-00	Time 10:13			
Fractional Wt Loss	? 	0.693	0.553	0.483	_	_	0.373	0.350	0.330	0.313			0.271	0.260	0.250		0.232	0.224		0.209	0.203	0.197	0.191	0.185	0.180		_	_			0.114	0.100	0.088	0.077	0.067	0.058	0.052	0.047	0.042
Wt. Loss, %	Ľ	4.323	3.450	3.014	2.721	2.501	2.326	2.181	2.058	1.949	1.853	1.769	1.693	1.623	1.560	1.502	1.447	1.397	1.350	1.306	1.265	1.228	1.191	1.157	1.124	1.093	1.063	1.035	0.948	0.818	0.711	0.623	0.548	0.480	0.416	0.365	0.324	0.291	0.263
SQRT Adj. Wt. Loss,	, G	45.57	72.65	92.09	108.08	122.00	134.49	145.91	156.50	166.41	175.77	184.65	193.13	201.25	209.06	216.59	223.86	230.91	237.74	244.38	250.85	257.15	263.31	269.32	275.20	280.96	286.61	292.14	310.72	340.22	367.35	392.62	416.35	438.81	460.17	480.58	500.16	519.00	537.18
Adjusted Time hr	ļш	0.577	1.466	2.356	3.245	4.135	5.024	5.914	6.803	7.693	8.582	9.471	10.361	11.251	12.141	13.031	13.921	14.810	15.700	16.589	17.479	18.368	19.259	20.148	21.038	21.928	22.818	23.707	26.818	32.152	37.486	42.820	48.153	53.487	58.821	64.154	69.488	74.822	80.156
Adjusted Time sec	<u>.</u> ш	2076.300	5278.380	8479.980	11680.740	14884.380	18086.340	21289.860	24491.880	27693.540	30894.360	34096.320	37299.120	40503.540	43707.660	46910.820	50115.240	53317.200	56519.100	59720.820	62923.380	66126.240	69330.660	72533.640	75735.840	78941.220	82143.300	85344.780	96545.700	115747.380	134949.360	154150.860	173351.220	192553.920	211754.460	230955.180	250157.220	269358.360	288559.980
TGA Time,	g 0	1596.300	4798.380	7999.980	11200.740	14404.380	17606.340	20809.860	24011.880	27213.540	30414.360	33616.320	36819.120	40023.540	43227.660	46430.820	49635.240	52837.200	56039.100	59240.820	62443.380	65646.240	68850.660	72053.640	75255.840	78461.220	81663.300	84864.780	96065.700	115267.380	134469.360	153670.860	172871.220	192073.920	211274.460	230475.180	249677.220	268878.360	288079.980
TGA mg	) ()	94.957	94.162	93.765	93.499	93.298	93.139	93.007	92.895	95.796	92.709	92.632	92.563	92.499	92.442	92.389	92.339	92.294	92.251	92.211	92.174	92.139	92.106	92.075	92.045	92.017	91.989	91.964	91.885	91.767	91.670	91.589	91.521	91,459	91.400	91.354	91.317	91.287	91.261
Temp.,	) (1) (2) (3) (4) (4) (4) (4) (4) (4) (4) (4) (4) (4	29.999	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30,000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000
TGA Time,	∢	26.605	79.973	133.333	186.679	240.073	293.439	346.831	400.198	453.559	506.906	560.272	613.652	667.059	720.461	773.847	827.254	880.620	933.985	987.347	1040.723	1094.104	1147.511	1200.894	1254.264	1307.687	1361.055	1414.413	1601.095	1921.123	2241.156	2561.181	2881.187	3201.232	3521.241	3841.253	4161.287	4481.306	4801.333

Blank

Supercritical Carbon Dioxide and

Polymethyl Methacrylate

**PMMA** 

### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

01-1-1

TGA Filename:

pmma075z

Material Name:

Property Test Specification: ASTM D471/D543

Material Code:

**PMMA** 

Material Supplier:

Polymethyl Methacrylate Harbor City Plastics

Material Class:

TP

Monomer Source:

Rohm & Haas

Base Polymer (Lot. No.): Plexiglass (76B818)

Replicate Number:

**Exposure Liquid:** 

Supercritical Carbon Dioxide

### **Pre-Exposure Data**

Measured Diameter (mm):

Initial Durometer Hardness:

<u>10.32</u>

10.29

10.30

0.087

Mean Diameter: Measured Thickness (mils):

38.30

<u>10.41</u>

38.90

38.20

38.20

Mean Thickness:

38.400

95% Confidence Level (CL):

D

95% Confidence Level (CL):

0.536

Initial Sample Weight (mg):

10.330

90.900 80.2

00:15:00

Scale: SHORE

Sample Exposure Data

**Exposure Date:** 

03/15/00

Time:

11:12:00 11:27:00

Removal Date:

03/15/00

Time:

Weight of Sample After Exposure (mg):

Total Exposure Time (h:m:s):

96.20

Weight Gain (mg):

5.30

Percent Weight Gain:

5.83

Final Durometer Hardness After Desorption:

79.5

Percent Hardness Change:

-0.9

#### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C): Time Lapse Between Sample Exposure and Sample Desorption (sec): 30.000

95% CL:

0.0012

Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

480

Sample Extracted (mg): Sample Extracted (%):

-0.82 -0.90 Solubility Corrected for Extractables (mg): Solubility Corrected for Extractables (%):

91.720 5.30 5.83

Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm²/sec):

Range Including Intercept (0,1), Relative Weight Fraction:

9.5093E-08 1.00 - 0.70

-7.1350E-03

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.84 - 0.701.3810E+02

Slope: R Square:

0.99602

Y-axis Intercept:

9.8532E-01

Low Concentration Diffusion Coefficient (cm²/sec):

1.8087E-09

Slope: -9.8402E-04

Linear Range of Desorption, Relative Weight Fraction:

0.20 - 0.15

R Square:

0.99931

X-axis Intercept: Y-axis Intercept:

4.4025E+02 4.3322E-01

Wt. @ Zero Time, mg K 96.20	Wt. @ Zero Time, % L 5.831	Adjustment to TGA Time, sec M 480	Hardness Shore Scale N D
TGA Dry Wt., mg 90.90	Equil. Wt. Of Desorb. Sample 91.72	Min. Wt. Used For FDC, mg. 90.90	Pre Exposure Hardness 80.2
High Conc. DC, cm2/sec 9.5093E-08	Low Conc. DC, cm2/sec 1.8087E-09	Total Liquid Sorbed, mg. 5.300	Post Desorption Hardness 79.5
R Square 0.99602	R Square 0.99931		Change in Hardness, % -0.9
X Intercept 138.10	X Intercept 440.3		Mean Thickness, cm.
Y Intercept 0.98532	Y Intercept 0.43322		0.0975
Y Intercept as Wt., mg. 94.788	X Intercept as Time, hrs. 53.8	X Intercept as Time, min. 323.0	pmma075z X Intercept as Time, days 2.2
TEMPERATURE STATIS		Max. TGA Wt., (orig data file) 95.283	TGA Wt. At 20 min. Adjustment 94.810
Column1		0 " 0	
Mean Standard Error Median	30.00002503 0.000611564 30	Sorption @ 7-12 min., mg 4.38	Sorption @ 20 min., mg 3.91
Mode Standard Deviation Sample Variance	30 0.031642147 0.001001225	Sorption @ 7-12 min., % 4.82	Sorption @ 20 min., % 4.30
Kurtosis Skewness Range	388.3126273 -12.72037019 1.251	Rel. Error (%) of 7-12 min. Sorp.	Rel. Error (%) of 20 min. Sorp
Minimum Maximum Sum Count	29.078 30.329 80310.067	-17.3	-26.2
Confidence Level(95.0%)	2677 0.001199186		

## pmma075z

## HIGH CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

Regression Statistics										
Multiple R	0.998007353									
R Square	0.996018676									
Adjusted R Square	0.995916591									
Standard Error	0.003392701									
Observations	41									

## ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.112304144	0.112304144	9756.73689	2.01829E-48
Residual	39	0.000448906	1.15104E-05		
Total	40	0.11275305			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.985321378	0.002340215	421.0388357	6.0118E-73	0.980587851	0.990054905
X Variable 1	-0.007135014	7.22341E-05	-98.77619599	2.0183E-48	-0.007281121	-0.006988907

## LOW CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

Regression Statistics				
Multiple R	0.999653993			
R Square	0.999308105			
Adjusted R Square	0.999307256			
Standard Error	0.000351402			
Observations	817			

### **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.145353831	0.145353831	1177109.34	0
Residual	815	0.000100639	1.23484E-07		
Total	816	0.14545447			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.433216228	0.000237547	1823.708373	0	0.432749951	0.433682504
X Variable 1	-0.000984018	9.06974E-07	-1084.946701	0	-0.000985798	-0.000982238

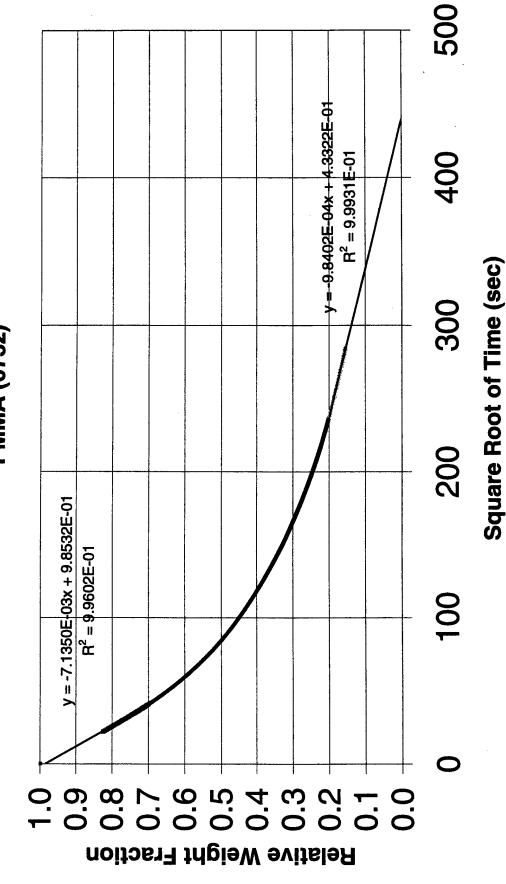
Column1				
Mean	10.33			
Standard Error	0.02738613			
Median	10.31			
Mode	#N/A			
Standard Deviation	0.05477226			
Sample Variance	0.003			
Kurtosis	2.92222222			
Skewness	1.70402573			
Range	0.12			
Minimum	10.29			
Maximum	10.41			
Sum	41.32			
Count	4			
Confidence Level(95.0%)	0.08715496			

## **THICKNESS STATISTICS**

	Column1			
	Mean	38.4		
	Standard Error	0.16832508		
	Median	38.25		
	Mode	38.2		
	Standard Deviation	0.33665016		
	Sample Variance	0.11333333		
	Kurtosis	3.57612457		
Plexiglass (76B818)	Skewness	1.88710473		
pmma075z	Range	0.7		
	Minimum	38.2		
	Maximum	38.9		
	Sum	153.6		
	Count	4		
	Confidence Level(95.0%)	0.53568604		

ND/value ND/value A/D

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: **PMMA (075z)** 



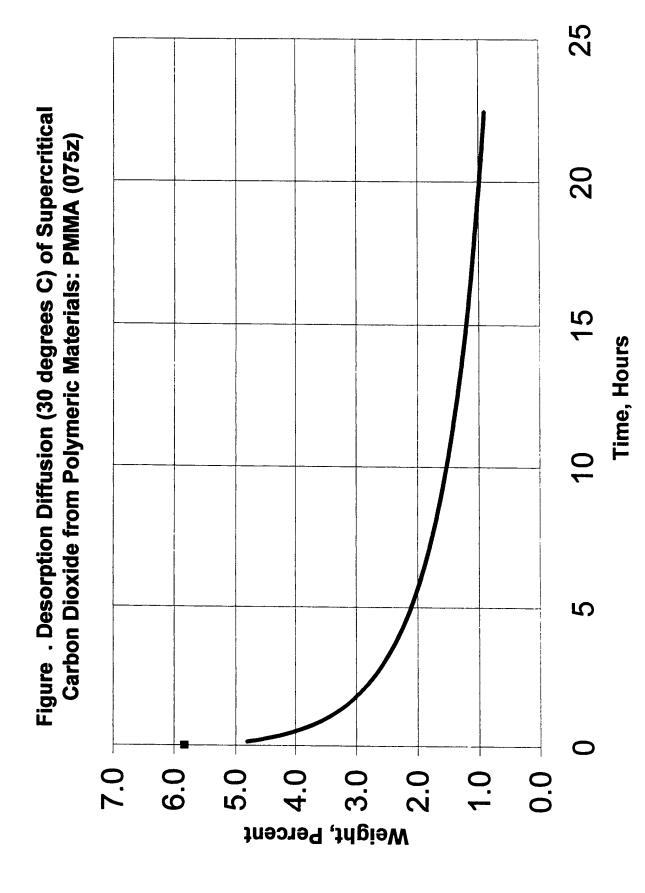


Figure . Desorption Diffusion (30 degrees C) of Supercritical

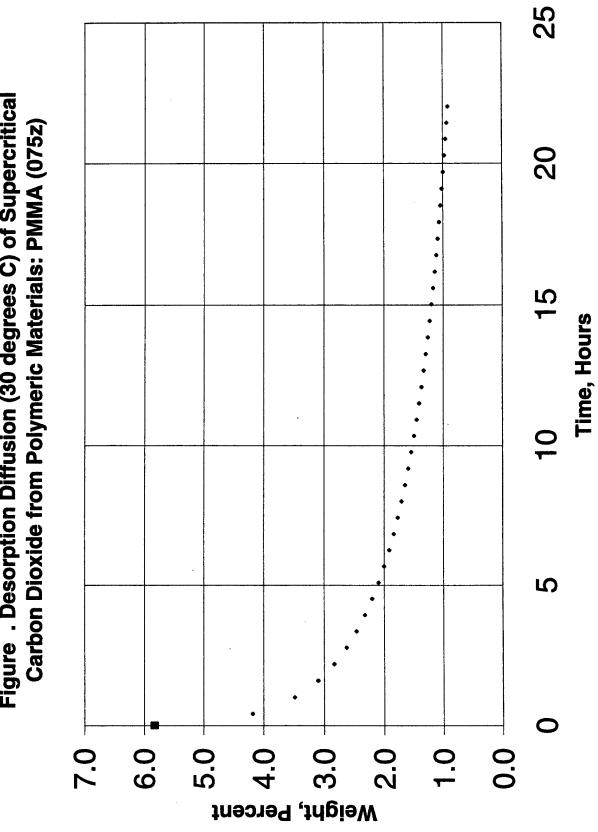
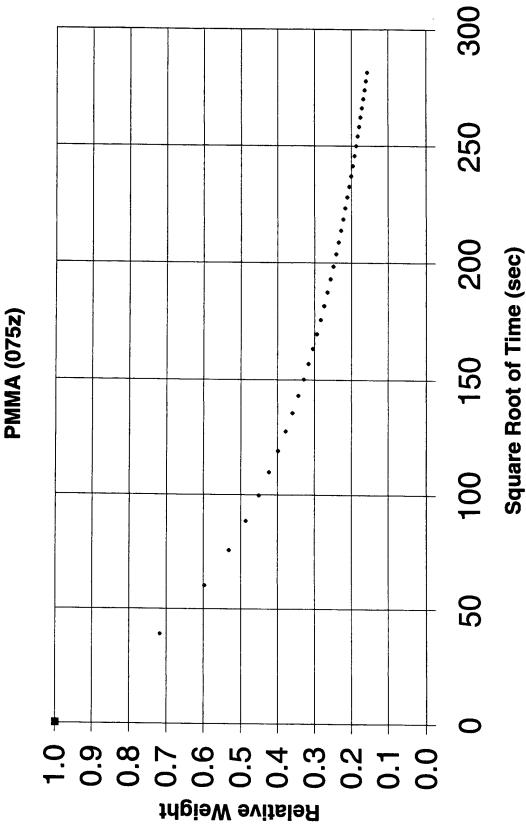


Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



Hardness	Scale	z	Shore D		Pre Exposure	Hardness	80.2		Post Desorption	Hardness	79.5		Change in	Hardness, %	-0.87															•										
Adjustment to	TGA Time, sec	Σ	480					Ā																															•	wt.
TGA Dry	Wt., mg #	_	6.06			of Data	lumns	eginning with	4								Q Q			omma075z	<del>Š</del>					8		ermal		ng m									•	ain dry sample
Wt. @ Zero	Time, %	¥	5.831			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4			<u>~</u>	ô	(ii	4) / L4) * 100)	/ (24 - L4))	1) / L4) * 100)	N16 = (((N12 - N8) / N8) * 100)			Source binary TA data file: pmma075z	14 lines in the parameter block	f data			000 aC	Sample PMMA sorbed in SCO2	Ď.	Method DESORPTION-isothermal		Comment Dry weight: 90.9 mg		<b>€</b>	ture (øC)	ng)		2			•	# Other balance used to obtain dry sample wt.
Wt. @ Zero	Time, mg	7	96.20			Formulae Use	Uppercase lett	Lowercase n r	***************************************	Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	In = ((Cn - L4) / (J4 - L4))	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 -			Source binary	14 lines in the	3 channels of data		Run 20	Module TGA 1000 øC	Sample PMM/	Size 95.283 mg	Method DESO	Operator BSI	Comment Dry	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 15-Mar-00	Time 11:35			# Other baland
Fractional	Wt. Loss		0.718	0.597	0.532	0.486	0.451	0.423	0.399	0.378	0.360	0.343	0.329	0.316	0.304	0.293	0.283	0.274	0.265	0.257	0.249	0.242	0.235	0.229	0.223	0.217	0.211	0.206	0.201	0.196	0.191	0.187	0.183	0.179	0.175	0.171	0.167	0.163	0.160	0.157
		I	4.184	3.482	3.100	2.836	2.632	2.466	2.325	2.203	2.097	2.002	1.918	1.842	1.774	1.71	1.652	1.598	1.546	1.498	1.453	1.410	1.370	1.333	1.297	1.264	1.231	1.201	1.172	1.143	1.116	1.090	1.066	1.043	1.019	0.996	0.975	0.953	0.933	0.913
SORT Adi. Wt. Loss.	Time, sec	Ø	39.07	60.23	75.70	88.50	99.66	109.70	118.90	127.43	135.43	142.98	150.15	156.99	163.55	169.85	175.94	181.81	187.50	193.03	198.40	203.62	208.72	213.69	218.56	223.31	227.98	232.55	237.03	241.42	245.73	249.97	254.15	258.25	262.28	266.26	270.18	274.04	277.85	281.61
Adjusted		u.	0.424	1.008	1.592	2.176	2.759	3.343	3.927	4.511	5.095	5.679	6.263	6.846	7.430	8.014	8.598	9.182	9.766	10.350	10.934	11.517	12.101	12.684	13.269	13.853	14.437	15.021	15.606	16.190	16.774	17.358	17.942	18.525	19.109	19.693	20.277	20.860	21.444	22.029
Adiusted	Time, sec	ш	1526.460	3627.840	5730.900	7831.860	9932.700	12034.320	14136.840	16238.040	18341.640	20443.920	22545.120	24645.960	26748.660	28850.460	30953.640	33055.080	35156.700	37259.580	39360.900	41461.740	43563.060	45663.960	47766.780	49869.000	51973.260	54077.220	56181.000	58284.300	60385.500	62487.120	64589.700	66691.260	68792.340	70895.460	72996.120	75096.540	77198.640	79303.260
TGA Time.	Sec	۵	1046.460	3147.840	5250.900	7351.860	9452.700	11554.320	13656.840	15758.040	17861.640	19963.920	22065.120	24165.960	26268.660	28370.460	30473.640	32575.080	34676.700	36779.580	38880.900	40981.740	43083.060	45183.960	47286.780	49389.000	51493.260	53597.220	55701.000	57804.300	59905.500	62007.120	64109.700	66211.260	68312.340	70415.460	72516.120	74616.540	76718.640	78823.260
TGA	Wt., mg	ပ	94.703	94.065	93.718	93.478	93.293	93.142	93.013	92.902	92.806	92.720	92.643	92.575	92.513	92.455	92.402	92.352	92.306	92.261	92.221	92.182	92.146	92.111	92.079	92.049	92.019	91.992	91.965	91.939	91.915	91.891	91.869	91.848	91.827	91.806	91.786	91.767	91.748	91.730
Temp	deg	ω	29.995	30.001	30.00	30.000	30.000	30.000	30.000	30.000	30.001	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30,000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000
TGA Time	nim	∢	17.441	52.464	87.515	122.531	157.545	192.572	227.614	262.634	297.694	332.732	367.752	402.766	437.811	472.841	507.894	542.918	577.945	612.993	648.015	683.029	718.051	753.066	788.113	823.150	858.221	893.287	928.350	963.405	998.425	1033.452	1068,495	1103.521	1138.539	1173.591	1208.602	1243.609	1278.644	1313.721

#### FIGURE . CHEMICAL-MATERIAL INTERACTION DATA SET:

**Supercritical Carbon Dioxide** Polyphenylene Oxide and

**PPO** 

#### Sorption, Desorption Diffusion, and Indentation Data Sets

#### Material Data

Test Reference Number: TGA Filename: 02-1-1 ppo0091d

Property Test Specification: ASTM D471/D543

Material Name: Polyphenylene Oxide Material Code: PPO Material Supplier: **Harbor City Plastics** Material Class: TP

Monomer Source: General Electric Base Polymer (Lot. No.): Noryl (NC8781)

Exposure Liquid: Supercritical Carbon Dioxide Replicate Number: 1

**Pre-Exposure Data** 

Measured Diameter (mm): 10.14 10.03 10.01 10.08

Mean Diameter: 10.065 95% Confidence Level (CL): 0.092

Measured Thickness (mils): 35.10 **36.10** 34.30 34.90

Mean Thickness: 35.100 95% Confidence Level (CL): 1.191

Initial Sample Weight (mg): 74.200

Initial Durometer Hardness: 76.2 Scale: SHORE D

Sample Exposure Data

**Exposure Date:** 03/31/00 Time: 14:12:00 Removal Date: 03/31/00 Time: 14:27:00

Total Exposure Time (h:m:s): 00:15:00 Weight of Sample After Exposure (mg): 76.90

Weight Gain (mg): 2.70 Percent Weight Gain: <u>3.64</u>

Final Durometer Hardness After Desorption: Percent Hardness Change: 76.6 0.5

**Desorption Experiment** 

Mean Temperature of Desorption Experiment (setpoint = 30 deg C): 30.000 95% CL: 0.0006

Time Lapse Between Sample Exposure and Sample Desorption (sec): 495 Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

74.380

Sample Extracted (mg): <u>-0.18</u> Solubility Corrected for Extractables (mg): 2.70 Sample Extracted (%): -0.24 Solubility Corrected for Extractables (%): <u>3.64</u>

**Diffusion Coefficient Data: Fick's Law** 

High Concentration Diffusion Coefficient (cm²/sec): 2.7634E-07

Range Including Intercept (0.1), Relative Weight Fraction: 1.00 - 0.55 Linear Range of Desorption, Relative Weight Fraction: 0.70 - 0.55

Slope: -1.3307E-02 X-axis Intercept: 7.4801E+01

R Square: 0.99852 Y-axis Intercept: 9.9534E-01

Low Concentration Diffusion Coefficient (cm²/sec): 1.6204E-10

Linear Range of Desorption, Relative Weight Fraction: 0.10 - 0.07Slope: -3.222E-04 X-axis Intercept: 4.6923E+02

R Square: 0.94367 Y-axis Intercept: 1.5119E-01

Wt. @ Zero	Wt. @ Zero	Adjustment to	Hardness
Time, mg	Time, %	TGA Time, sec	Shore Scale
K	L	M	N
76.90	3.639	495	D
TGA Dry	Equil. Wt. Of	Min. Wt. Used	Dro Evanous
Wt., mg	Desorb. Sample	For FDC, mg.	Pre Exposure Hardness
74.200	74.38	74.200	
74.200	74.30	74.200	76.2
High Conc.	Low Conc.	Total Liquid	Post Desorption
DC, cm2/sec	DC, cm2/sec	Sorbed, mg.	Hardness
2.7634E-07	1.6204E-10	2.700	76.6
R Square	R Square		Change in
0.99852	0.94367		Hardness, %
			0.5
X Intercept	X Intercept		
74.80	469.2		Mean Thickness,
			cm.
Y Intercept	Y Intercept		0.0892
0.99534	0.15119		
			ppo0091d
Y Intercept	X Intercept	X Intercept	X Intercept
as Wt., mg.	as Time, hrs.	as Time, min.	as Time, days
76.542	61.2	367.0	2.5
		May TCA MA	TCA 145 44.00
TEMPERATURE STATIS	TICE	Max. TGA Wt.,	TGA Wt. At 20
TEIM EIGHTORE STATIS	1103	(orig data file) 76.104	min. Adjustment
Column1		70.104	75.665
		Sorption @ 7-12	Sorption @ 20
Mean	30.0000646	min., mg	min., mg
Standard Error	0.000327842	1.90	1.47
Median	30		
Mode	30	Sorption @ 7-12	Sorption @ 20
Standard Deviation	0.01815015	min., %	min., %
Sample Variance	0.000329428	2.57	1.97
Kurtosis	568.6634763		
Skewness	-15.90073963	Rel. Error (%) of	Rel. Error (%) of
Range	0.83	7-12 min. Sorp.	20 min. Sorp
Minimum	29.389	-29.5	-45.7
Maximum	30.219		
Sum	91950.198		
Count	3065		
Confidence Level(95.0%)	0.000642813		
		ı	

# ppo0091d

# HIGH CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

Regression Statistics										
Multiple R	0.99925928									
R Square	0.998519109									
Adjusted R Square	0.998451796									
Standard Error	0.003572855									
Observations	24									

#### ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.189359346	0.189359346	14833.9224	1.26428E-32
Residual	22	0.000280836	1.27653E-05		
Total	23	0.189640182			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.995342921	0.003090509	322.0643683	6.5574E-42	0.98893359	1.001752251
X Variable 1	-0.013306511	0.000109254	-121.7945911	1.2643E-32	-0.01353309	-0.013079933

## LOW CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

Regression S	tatistics
Multiple R	0.971426552
R Square	0.943669547
Adjusted R Square	0.943624518
Standard Error	0.001953709
Observations	1253

## **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.079993303	0.079993303	20957.2359	0
Residual	1251	0.004775039	3.81698E-06		
Total	1252	0.084768343			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.151194785	0.000493793	306.1905226	0	0.150226031	0.152163539
X Variable 1	-0.00032222	2.2258E-06	-144.7661422	0	-0.000326587	-0.000317854

## **DIAMETER STATISTICS**

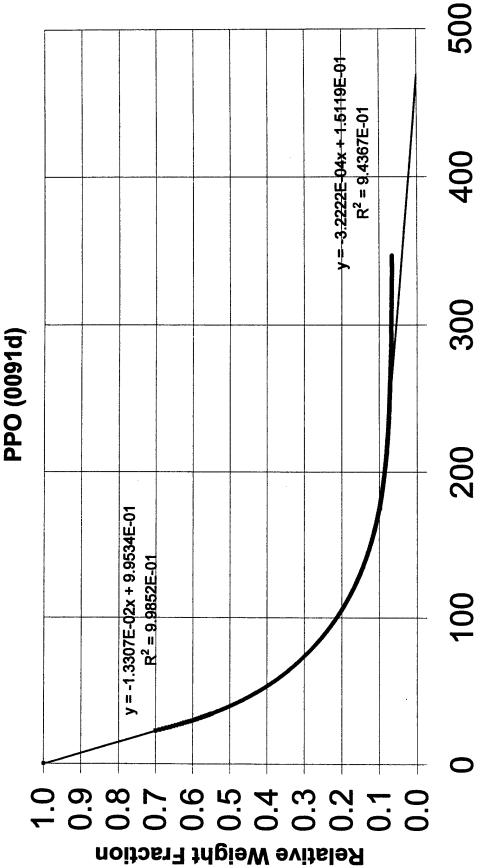
Column1								
Mean	10.065							
Standard Error	0.02901149							
Median	10.055							
Mode	#N/A							
Standard Deviation	0.05802298							
Sample Variance	0.00336667							
Kurtosis	-1.0475444							
Skewness	0.73716094							
Range	0.13							
Minimum	10.01							
Maximum	10.14							
Sum	40.26							
Count	4							
Confidence Level(95.0%)	0.0923276							

# THICKNESS STATISTICS

	Column1								
	Mean	35.1							
	Standard Error	0.37416574							
	Median	35							
	Mode	#N/A							
	Standard Deviation	0.74833148							
	Sample Variance	0.56							
	Kurtosis	1.5							
Noryl (NC8781)	Skewness	0.76360355							
ppo0091d	Range	1.8							
	Minimum	34.3							
	Maximum	36.1							
	Sum	140.4							
	Count	4							
	Confidence Level(95.0%)	1.19076349							

ND/value ND/value A/D

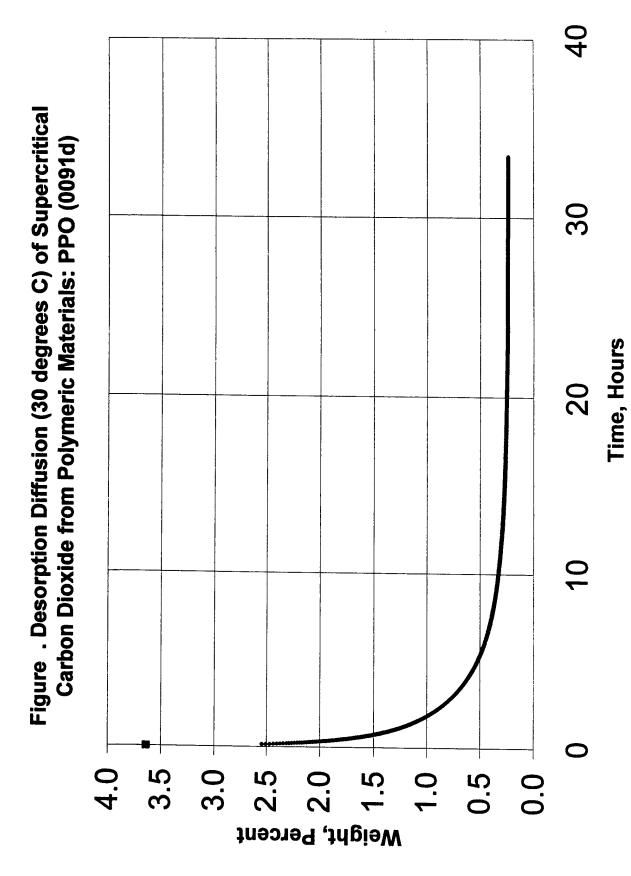
Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



Square Root of Time (sec)

APPENDIX B

82



80 Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PPO (0091d) 60 Time, Hours 40 20 3.5 2.5 0.5 Weight, Percent

900 Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PPO (0091d) 500 400 Square Root of Time (sec) 300 200 100 0.4 0.2 0.1 Relative Weight

Hardness Scale N Shore D	Pre Exposure	Hardness 76.2		Post Desorption	Hardness	76.6		Change in	Hardness, %	0.52															T: 8:00										
Adjustment to TGA Time, sec M	2		n=4																						Comment Disk: .4x0.03 in. HP@1400x50 Dry Wt, DS: 74.2 ET: 8:00										wt.
TGA Dry Wt., mg # L		of Data	peginning with									(O)			ppo0091d.01	ock		,		ø	•		hermal		1P@1400x50										ain dry sample
Wt. @ Zero Time, % K 3.639		Formulae Used for Analysis of Data Jopercase letters refer to columns	Lowercase n refers to row, beginning with n=4		_	₹	<u>6</u>	En)	Hn = (((Cn - L4)/L4) * 100)	)/(34-[4))	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8) /N8) * 100)			Source binary TA data file : ppo0091d.01	14 lines in the parameter block	of data			Module TGA Aluminum Pans	Sample PPO SCCO2 0091D	JG G	Method DESORPTION-Isothermal	"	k: .4x0.03 in. F		Œ	iture (øC)	mg)		8				# Other balance used to obtain dry sample wt.
Wt. @ Zero Time, mg J 76.90		Formulae Use Uppercase lef	Lowercase n		On = (An + 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hu = (((Cu - L	$\ln = ((Cn - L4) / (J4 - L4))$	K4 # ((()4 - L	N16 = (((N12			Source binary	14 lines in the	3 channels of data		Run 25	Module TGA	Sample PPO	Size 76.104 mg	Method DESC	Operator WJS	Comment Dis	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 31-Mar-00	Time 14:36			# Other balan
Fractional Wt. Loss I	0.315	0.202	0.151	0.134	0.121	0.112	0.10	0.097	0.092	0.088	0.085	0.081	0.079	0.077	0.075	0.074	0.073	0.072	0.071	0.071	0.00	0.069	0.069	0.068	0.068	0.067	0.067	990.0	990.0	990.0	990.0	990.0	0.065	0.065	0.065
Mt. Loss, % H	1.145	0.736	0.548	0.488	0.442	0.406	0.377	0.354	0.335	0.320	0.308	0.296	0.288	0.281	0.275	0.270	0.266	0.263	0.259	0.257	0.254	0.252	0.250	0.248	0.246	0.245	0.244	0.239	0.239	0.239	0.238	0.239	0.238	0.237	0.235
SQRT Adj. Wt. Loss, Time, sec % G H 44.61 1.741	70.64	104.88 118.33	130.41	141.46	151.70	161.29	170.34	178.95	187.15	195.01	202.57	209.85	216.89	223.71	230.32	236.75	243.01	249.11	255.07	260.89	266.58	272.16	277.63	282.99	288.24	293.99	315.89	343.20	368.50	392.16	414.48	435.65	455.84	475.18	493.75
Adjusted Time, hr F	1.386	3.055 3.890	4.724	5.559	6.392	7.226	8.060	8.895	9.729	10.564	11.398	12.233	13.067	13.901	14.735	15.570	16.403	17.237	18.072	18.906	19.741	20.576	21.410	22.245	23.079	24.008	27.719	32.719	37.719	42.719	47.719	52.720	57.720	62.720	67.720
Adjusted Time, sec E 1989,960	4990.560 7995.840	10999.080 14002.980	17006.700	20010.720	23011.440	26013.180	29017.200	32022.720	35025.480	38030.520	41033.340	44038.440	47041.020	50044.380	53047.560	56050.260	59052.540	62054.760	65058.900	68062.200	71067.180	74072.220	77076.900	80081.460	83084.760	86428.800	99788.460	117788.760	135788.760	153789.960	171790.080	189790.260		225791.460	243791.760
TGA Time, sec D 1494,960	4495.560 7500.840	10504.080	16511.700	19515.720	22516.440	25518.180	28522.200	31527.720	34530.480	37535.520	40538.340	43543.440	46546.020	49549.380	52552.560	55555.260	58557.540	61559.760	64563.900	67567.200	70572.180	73577.220	76581.900	79586.460	82589.760	85933.800	99293.460	117293.760	135293.760	153294.960	171295.080	189295.260	207295.620	225296.460	243296.760
TGA Wt., mg C 75.492	75.050 74.862	74.746 74.666	74.607	74.562	74.528	74.501	74.480	74.463	74.449	74.438	74.428	74.420	74.414	74.408	74.404	74.400	74.397	74.395	74.392	74.391	74.389	74.387	74.386	74.384	74.383	74.382	74.381	74.378	74.377	74.377	74.377	74.378	74.377	74.376	74.374
Temp., deg C B	30.000	30.000 30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000
TGA Time, min A 24.916	74.926 125.014	175.068 225.133	275.195	325.262	375.274	425.303	475.370	525.462	575.508	625.592	675.639	725.724	775.767	825.823	875.876	925.921	975.959	1025.996	1076.065	1126.120	1176.203	1226.287	1276.365	1326.441	1376.496	1432.230	1654.891	1954.896	2254.896	2554.916	2854.918	3154.921	3454.927	3754.941	4054.946

#### FIGURE . CHEMICAL-MATERIAL INTERACTION DATA SET:

**Supercritical Carbon Dioxide** 

and

Polyphenylene Oxide

PPO

#### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

02-1-2

TGA Filename:

ppo0096d

Property Test Specification: ASTM D471/D543

Material Code:

PPO

Material Name: Material Supplier:

Polyphenylene Oxide **Harbor City Plastics** 

Material Class:

TP

Monomer Source:

**General Electric** 

Base Polymer (Lot. No.): Noryl (NC8781)

Exposure Liquid:

Supercritical Carbon Dioxide

Replicate Number:

2

**Pre-Exposure Data** 

Measured Diameter (mm):

10.16

33.40

10.1 10.06 95% Confidence Level (CL):

10.18

0.088

Mean Diameter: Measured Thickness (mils):

10.125

34.00

33.70 33.20

Mean Thickness:

33.575

95% Confidence Level (CL):

0.557

Initial Sample Weight (mg): Initial Durometer Hardness: 69.900 75.6

Scale: SHORE

D

#### Sample Exposure Data

Exposure Date:

04/06/00

Time:

10:49:00

Removal Date:

04/06/00

Time:

11:04:00

Total Exposure Time (h:m:s):

00:15:00

Weight of Sample After Exposure (mg): Percent Weight Gain:

72.90

Weight Gain (mg):

3.00

**75.8** 

Percent Hardness Change:

4.29

Final Durometer Hardness After Desorption:

<u>0.3</u>

## **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C): Time Lapse Between Sample Exposure and Sample Desorption (sec): 30.000

95% CL:

0.0007

Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

Solubility Corrected for Extractables (mg):

46<u>5</u> 70.020

Sample Extracted (mg):

-0.12

3.00

Sample Extracted (%):

-0.17

Solubility Corrected for Extractables (%):

<u>4.29</u>

#### Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm²/sec):

3.2831E-07

Range Including Intercept (0,1), Relative Weight Fraction: Linear Range of Desorption, Relative Weight Fraction:

1.00 - 0.50 0.63 - 0.50

Slope: R Square: -1.5163E-02 0.98852

X-axis Intercept: Y-axis Intercept:

6.4395E+01 9.7639E-01

Low Concentration Diffusion Coefficient (cm²/sec):

5.0719E-11

Linear Range of Desorption, Relative Weight Fraction: Slope: -1.8846E-04

X-axis Intercept:

0.07 - 0.044.9037E+02

R Square:

0.86699

Y-axis Intercept:

9.2416E-02

Wt. @ Zero Time, mg K 72.90	Wt. @ Zero Time, % L 4.292	Adjustment to TGA Time, sec M 465	Hardness Shore Scale N D
TGA Dry Wt., mg 69.900	Equil. Wt. Of Desorb. Sample 70.02	Min. Wt. Used For FDC, mg. 69.900	Pre Exposure Hardness 75.6
High Conc. DC, cm2/sec 3.2831E-07	Low Conc. DC, cm2/sec 5.0719E-11	Total Liquid Sorbed, mg. 3.000	Post Desorption Hardness 75.8
R Square 0.98852	R Square 0.86699		Change in Hardness, % 0.3
X Intercept 64.39	X Intercept 490.4		Mean Thickness, cm.
Y Intercept 0.97639	Y Intercept 0.09242		0.0853
Y Intercept as Wt., mg. 71.179	X Intercept as Time, hrs. 66.8	X Intercept as Time, min. 400.8	ppo0096d X Intercept as Time, days 2.8
TEMPERATURE STATIS		Max. TGA Wt., (orig data file) 71.818	TGA Wt. At 20 min. Adjustment 71.335
Column1			
Mean Standard Error Median	30.00018117 0.000342847 30	Sorption @ 7-12 min., mg 1.92	Sorption @ 20 min., mg 1.43
Mode Standard Deviation Sample Variance Kurtosis	30.001 0.018665481 0.0003484 477.8445073	Sorption @ 7-12 min., % 2.74	Sorption @ 20 min., % 2.05
Skewness Range Minimum Maximum Sum Count	-13.7793311 0.815 29.414 30.229 88920.537	Rel. Error (%) of 7-12 min. Sorp. -36.1	Rel. Error (%) of 20 min. Sorp -52.2
Confidence Level(95.0%)	2964 0.000672242		

## ppo0096d

# HIGH CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

Regression Statistics								
Multiple R	0.994245135							
R Square	0.988523388							
Adjusted R Square	0.987949557							
Standard Error	0.011315319							
Observations	22							

#### ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.220565126	0.220565126	1722.67459	7.02123E-21
Residual	20	0.002560729	0.000128036		
Total	21	0.223125855			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.97639493	0.009936018	98.2682294	2.5088E-28	0.955668769	0.997121092
X Variable 1	-0.015162683	0.000365321	-41.50511524	7.0212E-21	-0.015924729	-0.014400638

#### LOW CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.93112234								
R Square	0.866988812								
Adjusted R Square	0.866922638								
Standard Error	0.002762891								
Observations	2012								

## **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.100011233	0.100011233	13101.5108	0
Residual	2010	0.015343465	7.63356E-06		
Total	2011	0.115354699			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.092415842	0.000394355	234.3466947	0	0.091642455	0.093189229
X Variable 1	-0.000188461	1.64649E-06	-114.4618313	0	-0.00019169	-0.000185232

# **DIAMETER STATISTICS**

Column1	
Mean	10.125
Standard Error	0.02753785
Median	
	10.13
Mode	#N/A
Standard Deviation	0.05507571
Sample Variance	0.00303333
Kurtosis	-3.032967
Skewness	-0.3232314
Range	0.12
Minimum	10.06
Maximum	10.18
Sum	40.5
Count	4
Confidence Level(95.0%)	0.08763782

# THICKNESS STATISTICS

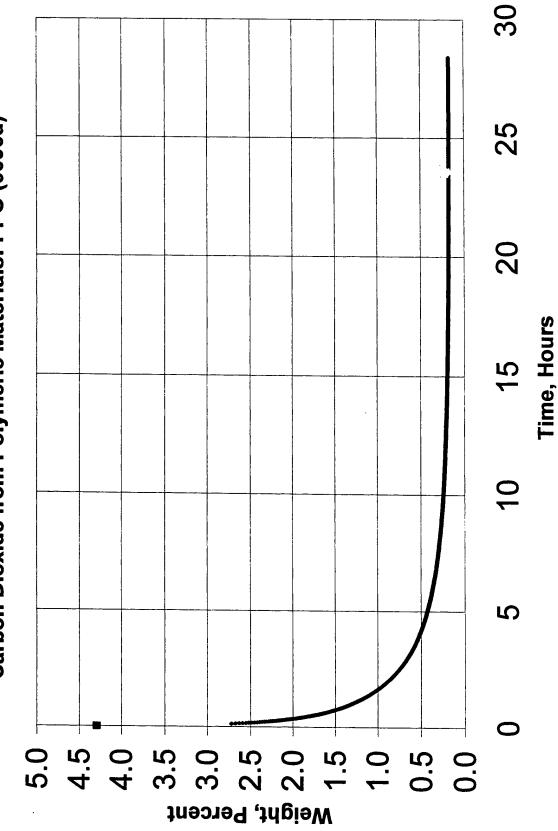
	Column1							
	Mean	33.575						
	Standard Error	0.175						
	Median	33.55						
	Mode	#N/A						
	Standard Deviation	0.35						
	Sample Variance	0.1225						
	Kurtosis	-1.5976676						
Noryl (NC8781)	Skewness	0.32069971						
ppo0096d	Range	0.8						
	Minimum	33.2						
	Maximum	34						
	Sum	134.3						
	Count	4						
	Confidence Level(95.0%)	0.55692863						

ND/value ND/value A/D

500 Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: 400 y = -1.8846E-04x + 9.2416E-02 $R^2 = 8.6699E-01$ 300 PPO (0096d) 200 y = -1.5163E-02x + 9.7639E-01 $R^2 = 9.8852E-01$ 100 0.6 0.5 0.3 0.3 Relative Weight Fraction

Square Root of Time (sec)

Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PPO (0096d)



Hardness	Scale	z	Shore D		Pre Exposure	Hardness	75.6		Post Desorption	Hardness	75.8		Change in	Hardness, %	0.26															T: 7:30	-									
Adjustment to	TGA Time, sec	≥	465					n=4																						Comment Disk: .4x0.03 in. HP@1400x50 Dry Wt, DS: 69.9 ET: 7:30									,	wt.
TGA Dry	Wt., mg #	_	4. 6.69			of Data	lumns	eginning with	1994111489345149								<del>Q</del>			po0096d.01	čķ				•			ermal		P@1400x50									•	ain dry sample
Wt. @ Zero	Time, %	¥	4.292			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4			•	6	Ē	t) / L4) * 100)	/ (74 - L4))	)/L4)*100)	N16 = (((N12 - N8) / N8) * 100)			Source binary TA data file: ppo0096d.01	14 lines in the parameter block	data	************		Module TGA Aluminum Pans	Sample PPO SCCO2 0096D	6	Method DESORPTION-Isothermal		c: .4x0.03 in. H		<u>ج</u>	ture (øC)	ng)		•			;	# Other balance used to obtain dry sample wt.
Wt. @ Zero	Time, mg	7	72.90			Formulae Use	Uppercase lett	Lowercase n r	****************	Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	In = ((Cn - L4) / (J4 - L4))	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 -			Source binary	14 lines in the	3 channels of data		Run 26	Module TGA A	Sample PPO S	Size 71.818 mg	Method DESO	Operator WJS	Comment Disk	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 6-Apr-00	Time 11:13			# Other baland
Fractional	Wt. Loss	_	0.449	0.298	0.229	0.185	0.154	0.131	0.114	0.101	0.091	0.083	0.076	0.071	0.067	0.063	0.059	0.057	0.055	0.053	0.051	0.049	0.048	0.047	0.046	0.046	0.045	0.044	0.044	0.043	0.043	0.042	0.042	0.042	0.042	0.042	0.041	0.041	0.0	0.041
		I	1.929	1.277	0.981	0.792	0.660	0.563	0.491	0.436	0.392	0.357	0.328	0.305	0.285	0.269	0.255	0.243	0.234	0.226	0.219	0.212	0.208	0.202	0.198	0.195	0.193	0.190	0.188	0.186	0.184	0.182	0.181	0.180	0.179	0.178	0.177	0.177	0.176	0.175
SQRT Adj. Wt. Loss,	Time, sec	g	40.14	62.55	78.82	92.28	104.01	114.55	124.20	133.14	141.53	149.44	156.95	164.12	171.00	177.60	183.97	190 13	196.10	201.89	207.52	212.99	218.32	223.54	228.63	233.61	238.49	243.27	247.96	252.56	257.09	261.52	265.89	270.18	274.41	278.58	282.68	286.72	290.71	296.67
Adjusted		L	0.448	1.087	1.726	2.365	3.005	3.645	4.285	4.924	5.564	6.203	6.843	7.482	8.122	8.762	9.402	10.041	10.682	11.322	11.962	12.601	13.241	13.881	14.520	15.160	15.799	16.439	17.079	17.719	18.359	18.999	19.638	20.277	20.917	21.557	22.197	22.836	23.476	24.448
Adjusted	Time, sec	ш	1611.240	3912.240	6213.240	8514.780	10817.280	13121.160	15424.980	17727.180	20029.980	22332.180	24634.620	26936.880	29239.380	31542.360	33846.300	36148.980	38453.640	40759.500	43062.480	45364.680	47665.800	49969.920	52273.080	54574.320	56877.960	59180.640	61484.400	63787.860	66093.120	68395.020	70696.560	72997.920	75302.520	77606.460	79908.120	82210.200	84512.700	88012.680
TGA Time,	sec	۵	1146.240	3447.240	5748.240	8049.780	10352.280	12656.160	14959.980	17262.180	19564.980	21867.180	24169.620	26471.880	28774.380	31077.360	33381.300	35683.980	37988.640	40294.500	42597.480	44899.680	47200.800	49504.920	51808.080	54109.320	56412.960	58715.640	61019.400	63322.860	65628.120	67930.020	70231.560	72532.920	74837.520	77141.460	79443.120	81745.200	84047.700	87547.680
TGA	Wt., mg	ပ	71.248	70.793	70.586	70.454	70.361	70.294	70.243	70.205	70.174	70.149	70.129	70.113	70.100	70.088	70.078	70.070	70.064	70.058	70.053	70.049	70.045	70.041	70.039	70.037	70.035	70.033	70.031	70.030	70.029	70.027	70.027	70.026	70.025	70.025	70.024	70.024	70.023	70.022
Temp.,	deg C	Δ	29.997	30.000	30.000	30.000	30.002	30.005	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000
TGA Time,	Ë	∢	19.104	57.454	95.804	134.163	172.538	210.936	249.333	287.703	326.083	364.453	402.827	441.198	479.573	517.956	556.355	594.733	633.144	671.575	709.958	748.328	786.680	825.082	863.468	901.822	940.216	978.594	1016.990	1055.381	1093.802	1132.167	1170.526	1208.882	1247.292	1285.691	1324.052	1362.420	1400.795	1459.128

Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PPO (0096d)

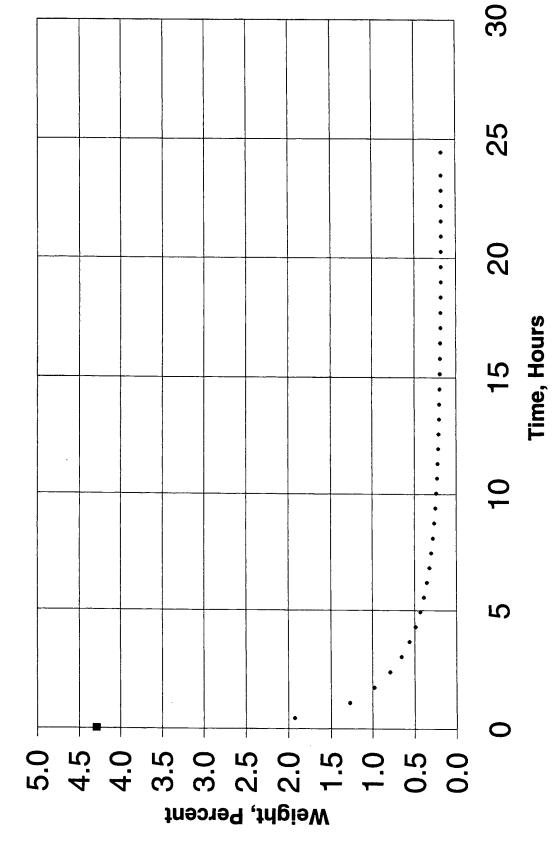
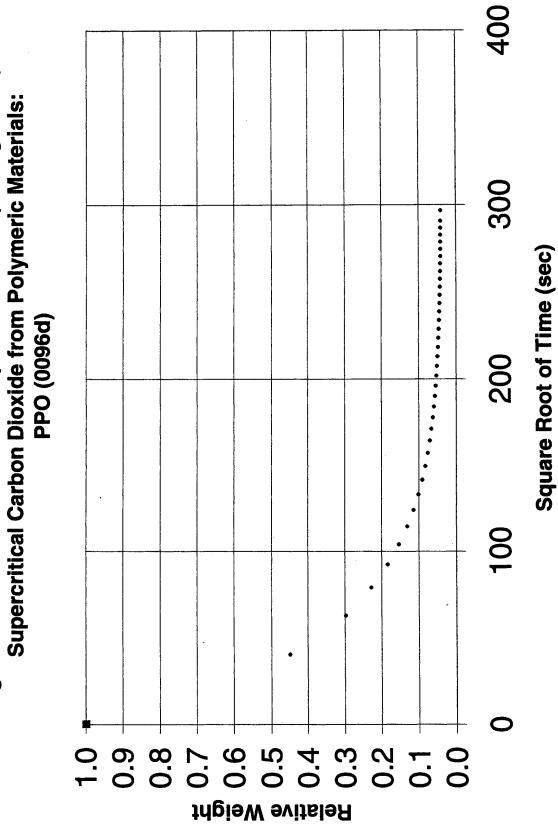


Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



#### FIGURE . CHEMICAL-MATERIAL INTERACTION DATA SET:

**Supercritical Carbon Dioxide** and

**Polyvinyl Chioride** 

**PVC** 

#### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

03-1-1

TGA Filename:

pvc0089d

Material Name:

Property Test Specification: ASTM D471/D543

Material Code:

**PVC** 

Material Supplier:

**Polyvinyl Chloride Harbor City Plastics** 

Material Class:

TP

Monomer Source:

Georgia Gulf

Base Polymer (Lot. No.): (4576T1)

**Exposure Liquid:** 

Supercritical Carbon Dioxide

Replicate Number:

Pre-Exposure Data

Measured Diameter (mm): Mean Diameter:

11.42

39.80

11.48

11.31

0.118

Measured Thickness (mils):

11.415

38.50

<u>41.00</u>

11.45

39.00

Mean Thickness: Initial Sample Weight (mg):

39.575

105.249

95% Confidence Level (CL):

1.735

Initial Durometer Hardness:

73.4

Scale: SHORE

D

95% Confidence Level (CL):

**Sample Exposure Data** 

**Exposure Date:** 

03/29/00

Time:

13:30:00

Removal Date:

03/29/00

Time:

13:45:00

Weight of Sample After Exposure (mg):

Total Exposure Time (h:m:s):

00:15:00

Percent Weight Gain:

106.80

Weight Gain (mg):

1.55

1.47

Final Durometer Hardness After Desorption:

Percent Hardness Change:

<u>-2.6</u>

71.5

**Desorption Experiment** 

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

Time Lapse Between Sample Exposure and Sample Desorption (sec):

30.001

95% CL:

0.0016

Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

Solubility Corrected for Extractables (mg):

660 105.280

Sample Extracted (mg):

-0.03

1.55

Sample Extracted (%):

-0.03

Solubility Corrected for Extractables (%):

1.47

Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm²/sec):

1.2426E-07

Range Including Intercept (0,1), Relative Weight Fraction:

Linear Range of Desorption, Relative Weight Fraction:

1.00 - 0.65 0.78 - 0.65

-7.9140E-03 Slope: R Square:

0.99783

X-axis Intercept: Y-axis Intercept:

1.2501E+02 9.8937E-01

Low Concentration Diffusion Coefficient (cm²/sec):

1.5727E-09

Linear Range of Desorption, Relative Weight Fraction: Slope: -8.9033E-04

X-axis Intercept:

0.07 - 0.02 3.2360E+02

R Square:

0.99847

Y-axis Intercept:

2.8812E-01

Wt. @ Zero	Wt. @ Zero	Adjustment to	Hardness				
Time, mg	Time, %	TGA Time, sec	Shore Scale				
K	L	M	N				
106.80	1.474	660	D				
TGA Dry	Equil. Wt. Of	Min. Wt. Used	Pre Exposure				
Wt., mg	Desorb. Sample	For FDC, mg.	Hardness				
105.249	105.28	105.249	73.4				
High Conc.	Low Conc.	Total Liquid	Post Desorption				
DC, cm2/sec	DC, cm2/sec	Sorbed, mg.	Hardness				
1.2426E-07	1.5727E-09	1.551	71.5				
R Square	R Square		Change in				
0.99783	0.99847		Hardness, % -2.6				
X Intercept	X Intercept		~2.0				
125.01	323.6		Mean Thickness,				
.20.0	020.0		cm.				
Y Intercept	Y Intercept		0.1005				
0.98937	0.28812		0.1000				
3.3223.			pvc0089d				
Y Intercept	X Intercept	X Intercept	X Intercept				
as Wt., mg.	as Time, hrs.	as Time, min.	as Time, days				
105.665	29.1	174.5	1.2				
		Max. TGA Wt.,	TGA Wt. At 20				
TEMPERATURE STATIST	rics	(orig data file)	min. Adjustment				
		106.469	106.354				
Column1		'					
Mean	00 00070000	Sorption @ 7-12	Sorption @ 20				
Standard Error	30.00078699	min., mg	min., mg				
Median	0.000801693	1.22	1.11				
Mode	30	Comtion @ 740	0				
	30	Sorption @ 7-12	Sorption @ 20				
Standard Deviation	0.043321102	min., %	min., %				
Sample Variance	0.001876718	1.16	1.05				
Kurtosis Skewness	256.1938329	Dal E (0/) -5	D.1 F (0/) . (				
	-6.545547698	· · ·	Rel. Error (%) of				
Range Minimum	1.567	7-12 min. Sorp.	20 min. Sorp				
	28.934	-21.3	-28.8				
Maximum Sum	30.501						
	87602.298						
Confidence Level(95.0%)	2920						
Confidence Level(95.0%)	0.00157194						

# **DIAMETER STATISTICS**

Column1	
Mean	11.415
Standard Error	0.03708099
Median	11.435
Mode	#N/A
Standard Deviation	0.07416198
Sample Variance	0.0055
Kurtosis	2.02644628
Skewness	-1.3729161
Range	0.17
Minimum	11.31
Maximum	11.48
Sum	45.66
Count	4
Confidence Level(95.0%)	0.11800838

ND/value ND/value A/D

# **THICKNESS STATISTICS**

	Column1	
	Mean	39.575
	Standard Error	0.54524459
	Median	39.4
	Mode	#N/A
	Standard Deviation	1.09048919
	Sample Variance	1.18916667
	Kurtosis	-0.3686457
(4576T1)	Skewness	0.75707136
pvc0089d	Range	2.5
	Minimum	38.5
	Maximum	41
	Sum	158.3
	Count	4
	Confidence Level(95.0%)	1.73521327

APPENDIX B

# HIGH CONCENTRATION DIFFUSION COEFFICIENT

pvc0089d

## **SUMMARY OUTPUT**

Regression Statistics													
Multiple R	0.998914632												
R Square	0.997830441												
Adjusted R Square	0.997777525												
Standard Error	0.002797958												
Observations	43												

# **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.147622177	0.147622177	18856.8519	3.08293E-56
Residual	41	0.000320971	7.82857E-06		
Total	42	0.147943149			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.989368347	0.002041735	484.572376	1.1317E-78	0.985244978	0.993491717
X Variable 1	-0.007914049	5.76321E-05	-137.3202531	3.0829E-56	-0.008030439	-0.007797659

# LOW CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

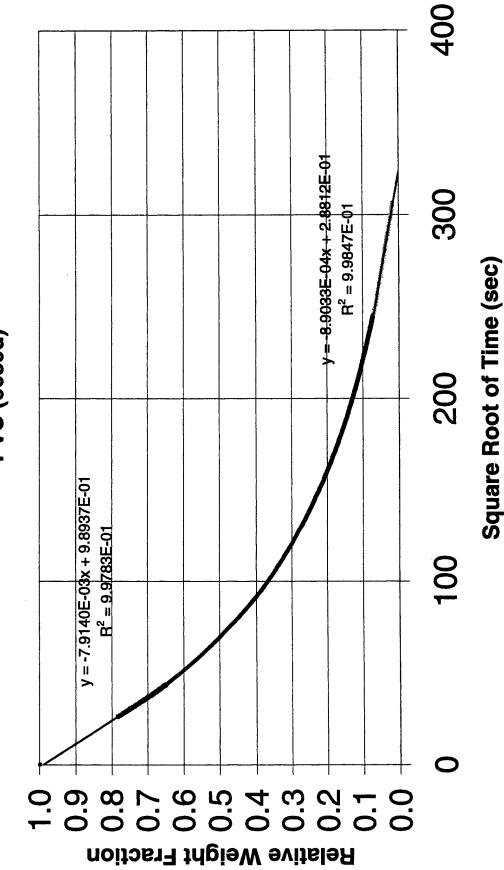
Regression Statistics													
Multiple R	0.999235225												
R Square	0.998471034												
Adjusted R Square	0.99846937												
Standard Error	0.000523843												
Observations	921												

## **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.164685731	0.164685731	600140.821	0
Residual	919	0.000252184	2.74412E-07		_
Total	920	0.164937915			

		Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.288115378	0.000313944	917.729431	0	0.287499249	0.288731508
X Variable 1	-0.000890331	1.14928E-06	-774.6875633	0	-0.000892587	-0.000888076

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PVC (0089d)



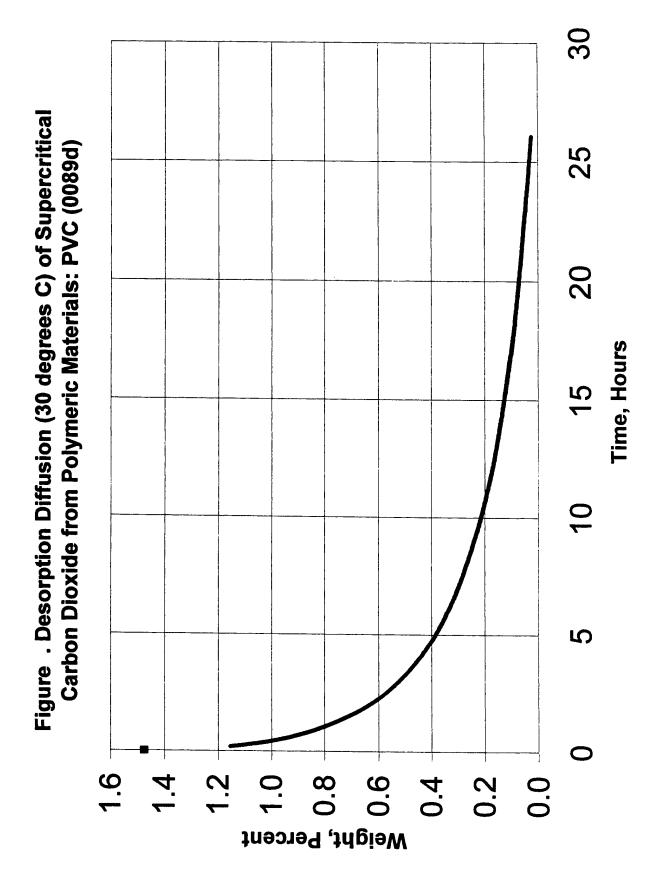


Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PVC (0089d)

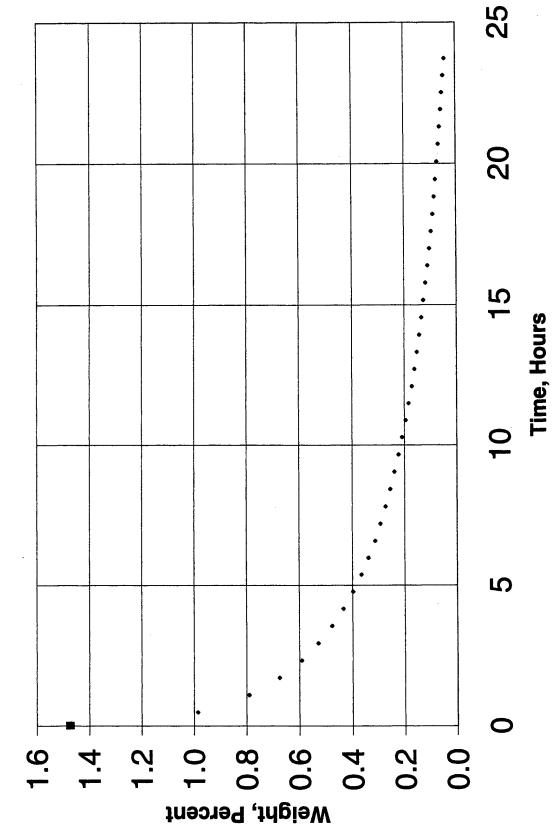
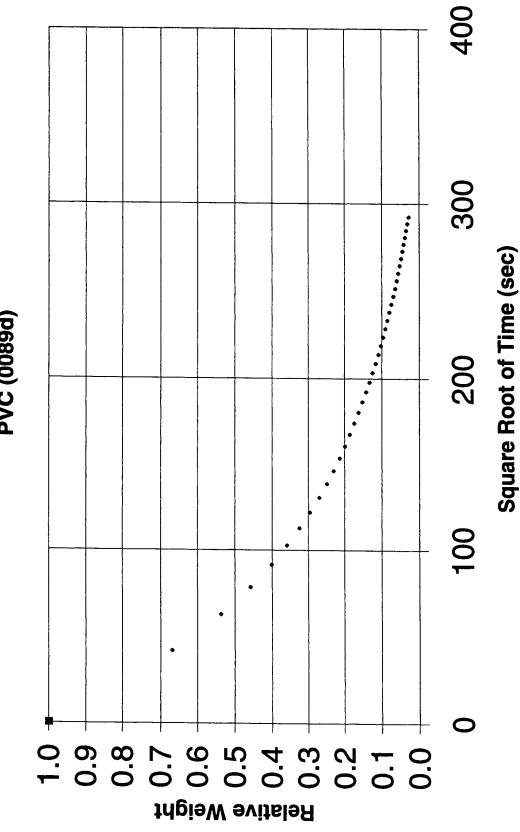


Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PVC (0089d)



Coordinate	Cale	D Z	Shore D		Pre Exposure	Hardness	73.4		Post Desorption	Hardness	71.5		Change in	Hardness, %	-2.59															49 ET: 11:30											
A discontaction &	Adjustment to	Des illies sec	099					n=4	1						•															Comment Disk: .4x0.03 in. HP @1400x50 Dry Wt, TGA: 105.249 E1: 11:30											
Č	Z	Wt., mg	105.249			of Data	lumns	eginning with									ĝ			ovc0089d.01	хķ				•			ermal		IP @ 1400x50											
140 0 7000	Wt. 69 2600	, % K. 6, %	1.474			d for Analysis	ers refer to co	efers to row, b			•	6	æ.	t) / L4) * 100)	/(34 - 1.4))	() / [-4] * 100)	N8) / N8) * 10			TA data file : p	14 lines in the parameter block	data			<b>Juminum Pan</b> e	3CCO2 0089D	ng	RPTION-Isoth		c: .4x0.03 in. H		2	ture (øC)	ng)		0					
M 6 72	Wi. 69 Zero Timo me	gm ,=	106.80			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4		Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	$\ln = ((Cn - L4) / (J4 - L4))$	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8) / N8) * 100)			Source binary TA data file : pvc0089d.01	14 lines in the	3 channels of data		Run 23	Module TGA Aluminum Pans	Sample PVC SCCO2 0089D	Size 106.469 mg	Method DESORPTION-Isothermal	Operator WJS	Comment Disk	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 29-Mar-00	Time 14:01				
	Fractional	WI. LOSS	0.669	0.536	0.458	0.402	0.359	0.324	0.295	0.270	0.249	0.230	0.213	0.199	0.186			0.152	0.142	0.133	0.124	0.116	0.109	0.103	960.0	0.00	0.085	0.079	_				-	0.052	0.048	0.044	0.04	0.038	0.035	0.032	0.028
1 47	Wt. Loss,	ደ ጋ	0.986	0.791	0.676	0.592	0.529	0.478	0.435	0.398	0.367	0.339	0.314	0.294	0.274	0.256	0.239	0.223	0.209	0.196	0.183	0.171	0.161	0.151	0.142	0.133	0.125	0.117	0.109	0.102	0.095	0.088	0.083	0.077	0.071	0.066	0.061	0.056	0.051	0.047	0.041
TO TO	Time and Wt. Loss, Fractional	) eec	41.89	62.89	78.46	91.42	102.75	112.96	122.32	131.01	139.17	146.87	154.19	161.17	167.87	174.31	180.51	186.51	192.32	197.96	203.45	208.79	214.00	219.08	224.05	228.91	233.67	238.33	242.91	247.40	251.81	256.15	260.41	264.60	268.73	272.80	276.80	280.75	284.65	288.49	292.29
A district	Adjusted Time hr	Ξ Έ Έ	0.488	1.099	1.710	2.322	2.933	3.544	4.156	4.768	5.380	5.992	6.604	7.215	7.828	8.440	9.051	9.662	10.274	10.885	11.497	12.109	12.721	13.333	13.944	14.555	15.167	15.779	16.390	17.002	17.614	18.225	18.837	19.449	20.060	20.672	21.283	21.895	22.507	23.119	23.731
7	Adjusted Time 600	1 III 16, 88C	1755.000	3955.260	6156.720	8357.700	10558.200	12759.180	14962.320	17164.620	19368.720	21571.080	23774.460	25975.260	28179.480	30382.500	32584.500	34784.820	36986.040	39187.620	41390.760	43592.880	45795.540	47997.240	50197.680	52399.260	54602.100	56803.320	59003.880	61205.520	63408.600	65611.320	67812.960	70014.600	72216.120	74418.180	76620.060	78822.660	81025.140	83227.620	85430.760
,	i GA Jime,	29C	1095.000	3295.260	5496.720	7697.700	9898.200	12099.180	14302.320	16504.620	18708.720	20911.080	23114.460	25315.260	27519,480	29722.500	31924.500	34124.820	36326.040	38527.620	40730.760	42932,880	45135.540	47337.240	49537.680	51739.260	53942.100	56143.320	58343.880	60545.520	62748.600	64951.320	67152.960	69354.600	71556.120	73758.180	75960.060	78162.660	80365.140	82567.620	84770.760
Ç	45 44 5 44	wt., mg	106.287	106.081	105.960	105.872	105.806	105.752	105.707	105.668	105.635	105.606	105.580	105.558	105.537	105.518	105.501	105.484	105.469	105.455	105.442	105.429	105.418	105.408	105.398	105.389	105.381	105.372	105.364	105.356	105.349	105.342	105.336	105.330	105.324	105.318	105.313	105.308	105.303	105.298	105.292
		) ရှိရှိ	30.026	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30,000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000
	GA IIMe,	E <	18.250	54.921	91.612	128.295	164.970	201.653	238.372	275.077	311.812	348.518	385.241	421.921	458.658	495.375	532.075	568.747	605.434	642.127	678.846	715.548	752.259	788.954	825.628	862.321	899.035	935.722	972.398	1009.092	1045.810	1082.522	1119.216	1155.910	1192.602	1229.303	1266.001	1302.711	1339.419	1376.127	1412.846

**Supercritical Carbon Dioxide** and **Polyvinyl Chloride** 

**PVC** 

#### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

03-1-3

TGA Filename:

pvc0110z

Property Test Specification: ASTM D471/D543

Polyvinyl Chloride

Material Code:

**PVC** 

Material Name: Material Supplier:

**Harbor City Plastics** 

Material Class:

TP

Monomer Source:

Georgia Gulf

Base Polymer (Lot. No.): (4576T1)

Exposure Liquid:

Replicate Number:

Supercritical Carbon Dioxide

### **Pre-Exposure Data**

Measured Diameter (mm):

11.01

11

11.07 10.99 95% Confidence Level (CL):

0.057

Mean Diameter: Measured Thickness (mils):

11.005 42.60

43.00

41.90 41.10

Mean Thickness:

42.150

95% Confidence Level (CL):

1.328

Initial Sample Weight (mg): Initial Durometer Hardness:

100.640 ND

Scale: SHORE

 $\mathbf{D}$ 

### Sample Exposure Data

**Exposure Date:** 

04/19/00

Time:

08:37:00

Removal Date:

04/19/00

Time:

08:52:00

Total Exposure Time (h:m:s):

00:15:00

Weight of Sample After Exposure (mg): Percent Weight Gain:

102.30

Weight Gain (mg):

1.66

1.65

Final Durometer Hardness After Desorption:

ND

Percent Hardness Change:

<u>ND</u>

### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.000

95% CL:

0.0007

Time Lapse Between Sample Exposure and Sample Desorption (sec): Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

450

-0.02

Solubility Corrected for Extractables (mg):

100.660 1.66

Sample Extracted (mg): Sample Extracted (%):

-0.02

Solubility Corrected for Extractables (%):

1.65

### Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm2/sec):

1.2995E-07

Range Including 0,1 Intercept, Relative Weight Fraction: Linear Range of Desorption, Relative Weight Fraction:

1.00 - 0.70 0.83 - 0.70

Slope:

R Square:

-7.5987E-03 0.99801

X-axis Intercept: Y-axis Intercept:

1.3033E+02 9.9033E-01

Low Concentration Diffusion Coefficient (cm2/sec):

3.2042E-10

Slope:

-3.7732E-04

Linear Range of Desorption, Relative Weight Fraction:

0.06 - 0.01

R Square:

0.99010

X-axis Intercept: Y-axis Intercept:

4.6480E+02 1.7538E-01

Wt. @ Zero Time, mg K 102.30	Wt. @ Zero Time, % L 1.649	Adjustment to TGA Time, sec M 450	Hardness Shore Scale N D
TGA Dry Wt., mg 100.640	Equil. Wt. Of Desorb. Sample 100.66	Min. Wt. Used For FDC, mg. 100.640	Pre Exposure Hardness ND
High Conc. DC, cm2/sec 1.2995E-07	Low Conc. DC, cm2/sec 3.2042E-10	Total Liquid Sorbed, mg. 1.660	Post Desorption Hardness ND
R Square 0.99801	R Square 0.99010		Change in Hardness, % ND
X Intercept 130.33 Y Intercept 0.99033	X Intercept 464.8 Y Intercept		Mean Thickness, cm. 0.1071
Y Intercept as Wt., mg. 101.310	0.17538  X Intercept as Time, hrs. 60.0	X Intercept as Time, min. 360.1	pvc0110z X Intercept as Time, days 2.5
TEMPERATURE STATIS		Max. TGA Wt., (orig data file) 102.014	TGA Wt. At 20 min. Adjustment 101.848
Mean Standard Error Median Mode Standard Deviation Sample Variance Kurtosis Skewness Range Minimum Maximum Sum Count Confidence Level(95.0%)	30.0000917 0.000342257 30 30 0.020155416 0.000406241 596.0945094 -16.45057779 0.948 29.291 30.239 104040.318 3468 0.000671045	min., % 1.37	Sorption @ 20 min., mg 1.21  Sorption @ 20 min., % 1.20  Rel. Error (%) of 20 min. Sorp -27.2

Column1										
Mean	11.0175									
Standard Error	0.01796988									
Median	11.005									
Mode	#N/A									
Standard Deviation	0.03593976									
Sample Variance	0.00129167									
Kurtosis	3.01436004									
Skewness	1.6963868									
Range	0.08									
Minimum	10.99									
Maximum	11.07									
Sum	44.07									
Count	4									
Confidence Level(95.0%)	0.05718824									

### **THICKNESS STATISTICS**

	Column1	
	Mean	42.15
	Standard Error	0.4173328
	Median	42.25
	Mode	#N/A
	Standard Deviation	0.8346656
	Sample Variance	0.69666667
	Kurtosis	-1.2583503
(4576T1)	Skewness	-0.5365585
pvc0110z	Range	1.9
•	Minimum	41.1
	Maximum	43
	Sum	168.6
	Count	4
	Confidence Level(95.0%)	1.32814048

ND/value ND/value A/D

pvc0110z

### **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.999006486								
R Square	0.998013959								
Adjusted R Square	0.997957215								
Standard Error	0.002478215								
Observations	37								

# ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.108017579	0.108017579	17587.9965	6.95037E-49
Residual	35	0.000214954	6.14155E-06		
Total	36	0.108232533			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.990325638	0.001773751	558.3229407	1.0143E-70	0.986724729	0.993926548
X Variable 1	-0.007598731	5.72971E-05	-132.6197438	6.9504E-49	-0.00771505	-0.007482411

# LOW CONCENTRATION DIFFUSION COEFFICIENT

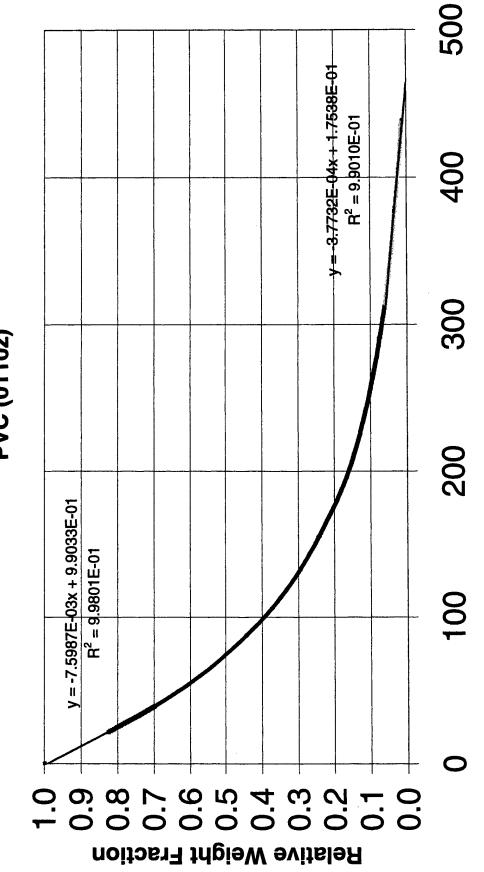
### **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.995040016								
R Square	0.990104633								
Adjusted R Square	0.990085785								
Standard Error	0.001369069								
Observations	527								

	df	SS	MS	F	Significance F
Regression	1	0.098459911	0.098459911	52530.1336	0
Residual	525	0.000984034	1.87435E-06		_
Total	526	0.099443945			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.175377777	0.000627973	279.2759937	0	0.17414413	0.176611424
X Variable 1	-0.000377322	1.6463E-06	-229.1945321	0	-0.000380556	-0.000374088

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PVC (0110z)



Square Root of Time (sec)

Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PVC (0110z)

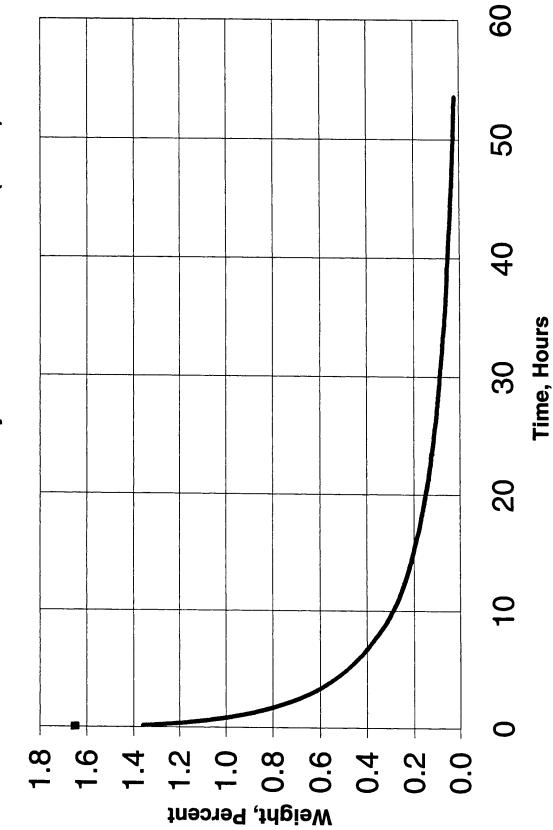


Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PVC (0110z)

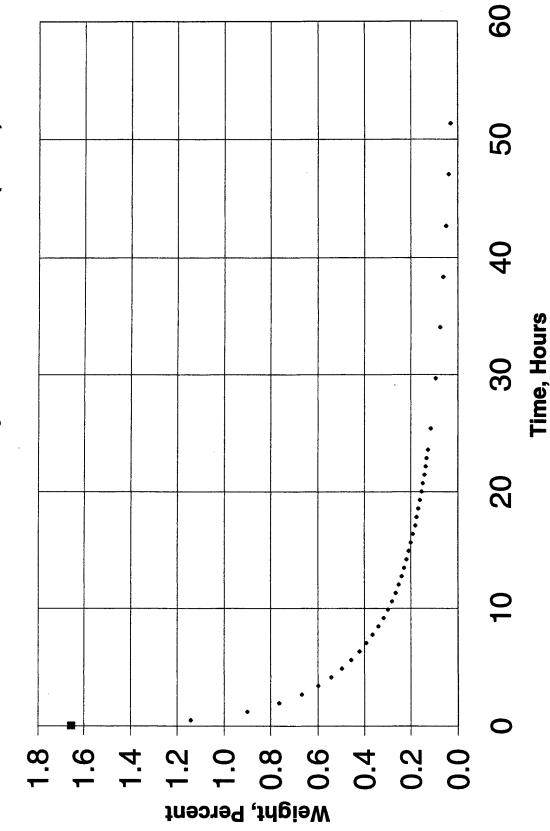
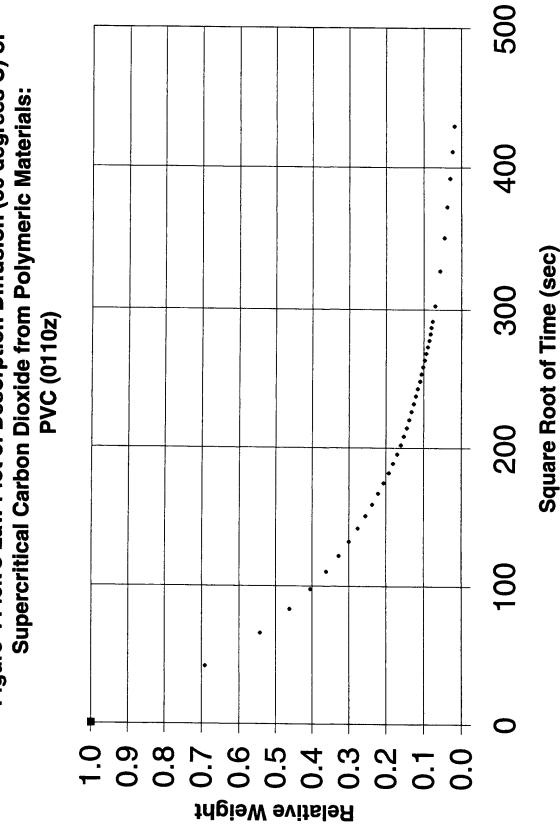


Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of



Hardness	ocaie N	Shore D		Pre Exposure	Hardness	2		Post Desorption	Hardness	2		Change in	Hardness, %	#VALUE!															00.635 ET-7:30												
Adjustment to	Sec allie, sec	450					4																						Comment Disk: .4x0.03 in., HP@1400x50, Dry: M-100.69 TG-100.635 ET-7:30												
TGA Dry	۸۲., mg	100.635			of Data	nmns	eginning with									6	•	•	vc0110z.01	¥					ZC		ərmal		P@1400x50,												
Wt. @ Zero	\$ • • •	1.654			for Analysis o	ers refer to col	fers to row, be				<u>~</u>	· E	)/L4)*100)	(74 - [4))	/L4) * 100)	N8) /N8) * 10(			FA data file: p	parameter blo	data	***************************************		uminum Pans	CO2 PVC011(	Đ.	<b>APTION-Isoth</b>		4x0.03 in., H		_	ure (øC)	(Bı								
Wt. @ Zero	ا الرام الرام	102.30			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4		Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	$\ln = ((Cn - L4) / (J4 - L4))$	K4 = (((J4 - L4)/L4) * 100)	N16 = (((N12 - N8) /N8) * 100)			Source binary TA data file: pvc0110z.01	14 lines in the parameter block	3 channels of data		Run 30	Module TGA Aluminum Pans	Sample PVC SCO2 PVC0110Z	Size 102.014 mg	Method DESORPTION-Isothermal	Operator WJS	Comment Disk:	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 19-Apr-00	Time 09:00					
Fractional	- 1	0.691	0.544	0.462	0.405	0.362	0.328	0.300	0.276	0.255	0.237	0.221	0.206	0.192	0.181	0.170	0.160	0.153	0.145	0.138	0.132	0.126				0.106		_	_		0.085			0.076	0.070	0.056	0.045	0.037	0.029	0.023	0.018
Wt. Loss,	Ľ	1.143	0.899	0.764	0.670	0.599	0.543	0.496	0.456	0.421	0.393	0.366	0.341	0.318	0.299	0.281	0.265	0.252	0.239	0.229	0.219	0.209	0.200	0.191	0.181	0.175	0.168	0.161	0.154	0.148	0.141	0.136	0.132	0.126	0.115	0.093	0.075	0.062	0.049	0.038	0.030
SQRT Adj. Wt. Loss, Time sec. %	G	41.77	65.92	83.34	97.70	110.25	121.48	131.76	141.29	150.23	158.67	166.68	174.31	181.62	188.66	195.44	201.99	208.33	214.48	220.46	226.30	231.98	237.52	242.94	248.24	253.43	258.51	263.49	268.38	273.19	277.91	282.55	287.12	291.61	302.66	326.94	349.99	371.60	392.03	411.45	429.99
Adjusted Time hr	Ē	0.485	1.207	1.929	2.652	3.376	4.099	4.822	5.545	6.269	6.993	7.717	8.440	9.163	9.886	10.610	11.333	12.056	12.778	13.501	14.225	14.948	15.672	16.395	17.117	17.841	18.563	19.286	20.008	20.731	21.454	22.177	22.899	23.622	25.445	29.692	34.025	38.358	42.692	47.025	51.359
Adjusted Time sec	<u>.</u> m	1744.980	4344.960	0945.300	9546.180	12154.020	14757.480	17360.100	19962.780	22568.280	25175.580	27781.800	30385.560	32987.640	35591.040	38195.580	40799.340	43401.720	46001.940	48604.140	51210.360	53814.240	56417.520	59020.260	61622.220	64226.160	66827.640	69429.480	72030.180	74631.000	77234.700	79835.460	82436.700	85038.060	91601.880	106889.940	122490.000	138090.060	153691.200		184891.200
TGA Time,	<u></u>	1294.980	3894.960	0485.300	9096.180	11704.020	14307.480	16910.100	19512.780	22118.280	24725.580	27331.800	29935.560	32537.640	35141.040	37745.580	40349.340	42951.720	45551.940	48154.140	50760.360	53364.240	55967.520	58570.260	61172.220	63776.160	66377.640	68979.480	71580.180	74181.000	76784.700	79385.460	81986.700	84588.060	91151.880	106439.940	122040.000	137640.060	153241.200	168841.080	184441.200
TGA W	ပ	101.785	101.540	404.101	101.309	101.238	101.181	101.134	101.094	101.059	101.030	101.003	100.978	100.955	100.936	100.918	100.902	100.889	100.876	100.865	100.855	100.845	100.836	100.827	100.817	100.811	100.804	100.797	100.790	100.784	100.777	100.772	100.768	100.762	100.751	100.729	100.710	100.697	100.684	100.673	100.665
Temp.,	B	29.998	30.000	30.000	30.000	30.000	30.000	30.00	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000
TGA Time, min	∢	21.583	64.916	100.233	151.603	195.067	238.458	281.835	325.213	368.638	412.093	455.530	498.926	542.294	585.684	629.093	672.489	715.862	759.199	802.569	846.006	889.404	932.792	976.171	1019.537	1062.936	1106.294	1149.658	1193.003	1236.350	1279.745	1323.091	1366.445	1409.801	1519.198	1773.999	2034.000	2294.001	2554.020	2814.018	3074.020

Blank

Supercritical Carbon Dioxide

and

Polyimide

PIF

### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

15-1-1

TGA Filename:

pif0151z

Material Name:

Property Test Specification: ASTM D471/D543

Material Code:

PIF

Material Supplier:

Polyimide Goodfellow Cambridge Ltd.

Material Class:

Blank

Monomer Source:

Unknown

Base Polymer (Lot. No.): Upilex

Exposure Liquid:

Supercritical Carbon Dioxide

Replicate Number:

1

### **Pre-Exposure Data**

Measured Diameter (mm): Mean Diameter:

8.42

8.42

8.55 8.44

0.099

Measured Thickness (mils):

8.430 10.00

10.800

95% Confidence Level (CL): 11.90 11.00

10.30

Mean Thickness:

19.260

95% Confidence Level (CL):

1.344

Initial Sample Weight (mg): Initial Durometer Hardness:

82.5

Scale: SHORE

D

### Sample Exposure Data

Exposure Date:

05/30/00

Time:

10:55:00

Removal Date:

05/30/00

Time:

11:10:00

Total Exposure Time (h:m:s):

00:15:00

Weight of Sample After Exposure (mg): Percent Weight Gain:

19.30

Weight Gain (mg):

0.04

0.21

Final Durometer Hardness After Desorption:

82.2

Percent Hardness Change:

<u>-0.4</u>

### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C): Time Lapse Between Sample Exposure and Sample Desorption (sec):

30.001

95% CL:

0.0005

Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

Solubility Corrected for Extractables (mg):

430 19.090

Sample Extracted (mg):

0.17

0.21

Sample Extracted (%):

0.88

Solubility Corrected for Extractables (%):

1.09

### Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm2/sec): Range Including 0,1 Intercept, Relative Weight Fraction:

1.8434E-09 1.00 - 0.90

Slope:

-3.5321E-03

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.95 - 0.90 2.8530E+02

R Square: 0.86086 Y-axis Intercept:

1.0077E+00

Low Concentration Diffusion Coefficient (cm2/sec):

1.1647E-10

Slope:

-8.8786E-04

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.05 - 0.004.0875E+02

R Square:

0.99548

Y-axis Intercept:

3.6291E-01

Wt. @ Zero Time, mg K 19.30	Wt. @ Zero Time, % L 0.208	Adjustment to TGA Time, sec M 430	Hardness Shore Scale N D
TGA Dry Wt., mg 19.260	Equil. Wt. Of Desorb. Sample 19.09	Min. Wt. Used For FDC, mg. 19.090	Pre Exposure Hardness 82.5
High Conc. DC, cm2/sec 1.8434E-09	Low Conc. DC, cm2/sec 1.1647E-10	Total Liquid Sorbed, mg. 0.040	Post Desorption Hardness 82.2
R Square 0.86086	R Square 0.99548		Change in Hardness, % -0.4
X Intercept 285.30	X Intercept 408.7		Mean Thickness, cm.
Y Intercept 1.00772	Y Intercept 0.36291		0.0274
Y Intercept as Wt., mg. 19.449	X Intercept as Time, hrs. 46.4	X Intercept as Time, min. 278.5	pif0151z X Intercept as Time, days 1.9
TEMPERATURE STATIS	rics	Max. TGA Wt., (orig data file) 19.291	TGA Wt. At 20 min. Adjustment 19.268
Column1			
Mean Standard Error	30.00114585 0.00026329	Sorption @ 7-12 min., mg	Sorption @ 20 min., mg
Median	30.001	0.03	0.01
Mode		Sorption @ 7-12	Sorption @ 20
Standard Deviation	0.015213991	min., %	min., %
Sample Variance	0.000231466	0.16	0.04
Kurtosis	693.6462665		0.0 .
Skewness	-20.3095439	Rel. Error (%) of	Rel. Error (%) of
Range	0.676	7-12 min. Sorp.	20 min. Sorp
Minimum	29.428	-22.5	-80.0
Maximum Sum	30.104		
Count	100173.826		
Confidence Level(95.0%)	3339 0.000516227		
	<del></del>		

Column1									
Mean	8.4575								
Standard Error	0.03119161								
Median	8.43								
Mode	8.42								
Standard Deviation	0.06238322								
Sample Variance	0.00389167								
Kurtosis	3.50405568								
Skewness	1.86901672								
Range	0.13								
Minimum	8.42								
Maximum	8.55								
Sum	33.83								
Count	4								
Confidence Level(95.0%)	0.09926572								

ND/value ND/value A/D

# **THICKNESS STATISTICS**

	Column1				
	Mean	10.8			
	Standard Error	0.42229532			
	Median	10.65			
	Mode	#N/A			
	Standard Deviation	0.84459063			
	Sample Variance	0.71333333			
	Kurtosis	-0.8058782			
pilex	Skewness	0.77679617			
if0151z	Range	1.9			
	Minimum	10			
	Maximum	11.9			
	Sum	43.2			
	Count	4			
	Confidence Level(95.0%)	1.34393343			

Up

pif0151z

### **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.927826825								
R Square	0.860862618								
Adjusted R Square	0.845402909								
Standard Error	0.011130957								
Observations	11								

### ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.006899181	0.006899181	55.68427	3.84247E-05
Residual	9	0.001115084	0.000123898		
Total	10	0.008014265			

		Standard Error		P-value	Lower 95%	Upper 95%
Intercept	1.00772173	0.010826876	93.07594323	9.6714E-15	0.983229616	1.032213845
X Variable 1						

# LOW CONCENTRATION DIFFUSION COEFFICIENT

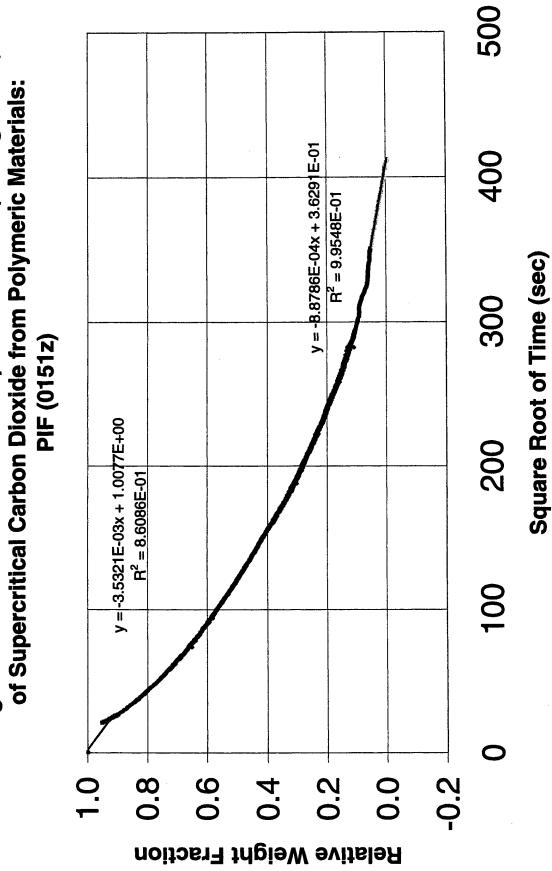
### **SUMMARY OUTPUT**

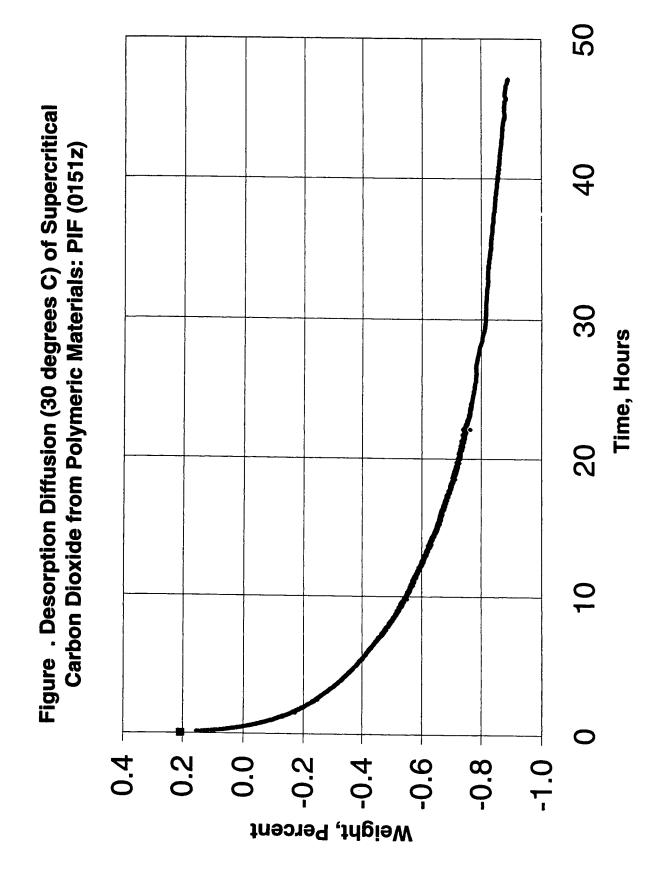
Regression Statistics									
Multiple R	0.997739896								
R Square	0.9954849								
Adjusted R Square	0.995467263								
Standard Error	0.001053154								
Observations	258								

	df	SS	MS	F	Significance F
Regression	1	0.062602448	0.062602448	56442.6332	3.1318E-302
Residual	256		1.10913E-06		
Total	257	0.062886387			

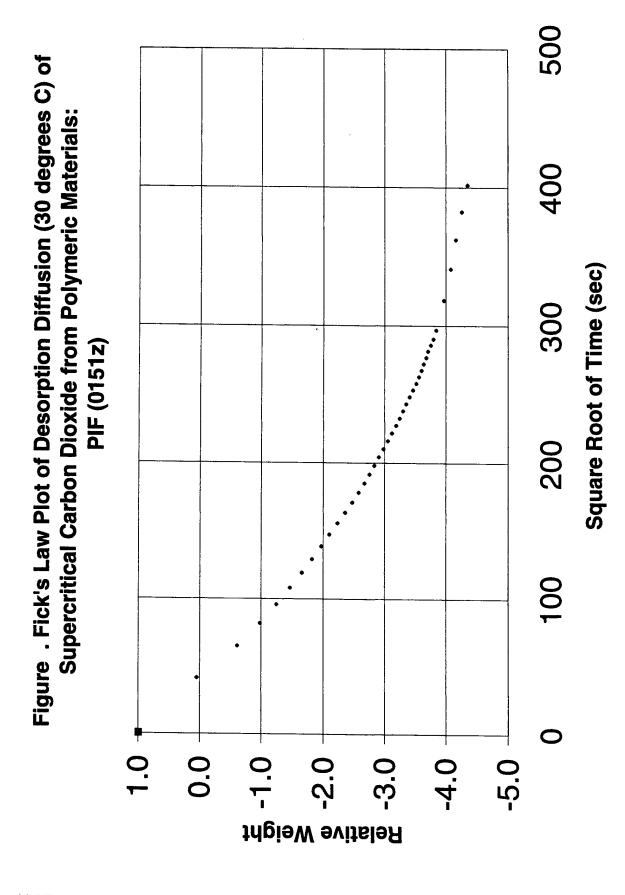
	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept		0.001430813			0.360091826	0.365727153
X Variable 1	-0.000887858	3.73714E-06	-237.5765839	3.132E-302	-0.000895217	-0.000880498

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:





50 Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PIF (0151z) 30 Time, Hours 0.2 <del>-</del>0.6 0.0 -0.4 <del>0</del>.0 0.4 Weight, Percent



Hardness Scale	z	Shore D	Pre Exposure	Hardness	82.5		Post Desorption	Hardness	82.2		Change in	Hardness, %	-0.36						٠									.261 ET: 7:10	÷											
Adjustment to TGA Time, sec	Z	430				th n=4	ł																					Comment .336"x.012", HP15@1400 Dry: M-19.36 TGA-19.261 E1: 7:10												
TGA Dry Wt., mg	)	19.261		of Data	olumns	beginning wil									<b>6</b>			pif0151z.01	lock		;		SI	0151Z		hermal		5@1400 Dry												
Wt. @ Zero Time. %	¥	0.202		d for Analysis	ters refer to co	efers to row,			•	6	į;	4) / L4) * 100)	/ (J4 - L4))	1) / L4) * 100)	- N8) /N8) * 1			TA data file :	14 lines in the parameter block	f data			<b>Numinum Par</b>	olyimide Film	Ď.	RPTION-Isot	!	3"x.012", HP1		(E)	ture (øC)	mg)		8						
Wt. @ Zero Time, ma	ر ب	19.30		Formulae Head for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4		Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	$\ln = ((Cn - L4) / (J4 - L4))$	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8) / N8) * 100)			Source binary TA data file: pif0151z.01	14 lines in the	3 channels of data		Run 46	Module TGA Aluminum Pans	Sample PIF Polyimide Film 0151Z	Size 19.291 mg	Method DESORPTION-Isothermal	Operator bsi	Comment .336	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 30-May-00	Time 11:18					
Fractional Wt. Loss	_	0.049	-0.608		_			-1.969			-2.356			-2.669	-2.754	-2.831	-2.903		-3.051	-3.115	-3.182					_		_			-3.674		_	-3.792	-3.838	-3.967	4.077	4.162	4.259	4.349
Wt. Loss, %	I	0.010	-0.123	25.6	-0.297	-0.336	-0.369	-0.399	-0.425	-0.452	-0.477	-0.500	-0.522	-0.540	-0.558	-0.573	-0.588	-0.603	-0.618	-0.631	-0.644	-0.657	-0.668	-0.678	-0.689	-0.700	-0.711	-0.720	-0.729	-0.736	-0.744	-0.750	-0.760	-0.768	-0.77	-0.803	-0.826	-0.843	-0.862	-0.881
SQRT Adj. Wt. Loss, Time, sec %	ග	40.96	64.66	05.00	108.09	119.11	129.18	138.53	147.29	155.55	163.40	170.89	178.07	184.97	191.62	198.04	204.26	210.29	216.16	221.89	227.46	232.89	238.21	243.41	248.50	253.49	258.38	263.17	267.89	272.52	277.07	281.55	285.97	290.31	296.83	318.25	341.00	362.33	382.47	401.60
Adjusted Time, hr	ш	0.466	1.161	2 551	3.245	3.941	4.635	5.331	6.026	6.721	7.416	8.112	8.808	9.504	10.199	10.894	11.589	12.284	12.980	13.676	14.371	15.067	15.762	16.457	17.153	17.849	18.544	19.239	19.934	20.630	21.325	22.020	22.716	23.411	24.474	28.134	32.301	36.468	40.634	44.801
Adjusted Time, sec	ш	1677.760	4180.960 6681.340	9182 140	11683.420	14186.500	16687.360	19190.920	21693.580	24195.940	26699.380	29204.440	31708.180	34213.900	36717.940	39219.760	41721.040	44223.460	46726.780	49233.040	51736.780	54239.980	56743.180	59246.620	61750.780	64254.820	66757.960	69260.260	71763.700	74266.900	76768.840	79272.520	81776.860	84280.660	88105.180	101283.460	116283.880	131283.940	146283.820	161283.880
TGA Time, sec	۵	1247.760	3750.960	8752 140	11253.420	13756.500	16257.360	18760.920	21263.580	23765.940	26269.380	28774.440	31278.180	33783.900	36287.940	38789.760	41291.040	43793.460	46296.780	48803.040	51306.780	53809.980	56313.180	58816.620	61320.780	63824.820	66327.960	68830.260	71333.700	73836.900	76338.840	78842.520	81346.860	83850.660	87675.180	100853.460	115853.880	130853.940	145853.820	160853.880
TGA Wt mg	, ပ	19.263	19.237	10.212	19.204	19.196	19.190	19.184	19.179	19.174	19.169	19.165	19.161	19.157	19.154	19.151	19.148	19.145	19.142	19.140	19.137	19.135	19.132	19.130	19.128	19.126	19.124	19.122	19.121	19.119	19.118	19.117	19.115	19.113	19.111	19.106	19.102	19.099	19.095	19.091
Temp., dea C	œ	29.992	30.002	50.00	30.00	30.003	30.005	30.000	30.001	30.002	30.002	30.003	30.002	30.002	30.000	30.005	30.002	30.002	30.005	30.002	30.001	30.001	30.005	30.001	30.002	30.000	30.001	30.000	30.000	30.000	30.000	30.005	30.002	30.002	30.001	30.002	30.002	30.002	30.005	30.002
'GA Time, min	⋖	20.796	62.516	14E B69	187.557	229.275	270.956	312.682	354.393	396.099	437.823	479.574	521.303	563.065	604.799	646.496	688.184	729.891	771.613	813.384	855.113	896.833	938.553	980.277	1022.013	1063.747	1105.466	1147.171	1188.895	1230.615	1272.314	1314.042	1355.781	1397.511	1461.253	1680.891	1930.898	2180.899	2430.897	2680.898

Blank

**Supercritical Carbon Dioxide** 

and

Acrylonitrile-butadiene-styrene

**ABS** 

### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

05-1-1

TGA Filename:

abs0115d

Property Test Specification: ASTM D471/D543

Material Code:

Material Name: Material Supplier: Acrylonitrile-butadiene-styrene Goodfellow

Material Class:

**ABS** TP

Monomer Source:

Base Polymer (Lot. No.): Unknown

Unknown

Exposure Liquid:

Supercritical Carbon Dioxide

Replicate Number:

1

### **Pre-Exposure Data**

Measured Diameter (mm): Mean Diameter:

10.48

10.65

10.32 10.51

0.215

Measured Thickness (mils):

10.495 65.60

95% Confidence Level (CL): 62.00 61.40

59.70

Mean Thickness:

62.175

138.450

95% Confidence Level (CL):

3.950

Initial Sample Weight (mg): Initial Durometer Hardness:

72.6

Scale: SHORE

D

### Sample Exposure Data

Exposure Date:

04/24/00

Time:

14:57:00 15:12:00

Removal Date: Total Exposure Time (h:m:s):

04/24/00

Time: 00:15:00

Weight of Sample After Exposure (mg):

143.60

Weight Gain (mg):

Percent Weight Gain:

3.72

Final Durometer Hardness After Desorption:

**73.4** 

Percent Hardness Change:

1.1

### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.000

95% CL:

0.0009

Time Lapse Between Sample Exposure and Sample Desorption (sec): Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

495

Sample Extracted (mg):

0.04

Solubility Corrected for Extractables (mg):

138.410

Sample Extracted (%):

0.03

Solubility Corrected for Extractables (%):

5.19 3.75

### Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm2/sec): Range Including 0.1 Intercept, Relative Weight Fraction:

5.2834E-07 1.00 - 0.63

-1.0387E-02

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.77 - 0.63

Slope: R Square:

0.99973

Y-axis Intercept:

9.6140E+01 9.9862E-01

Low Concentration Diffusion Coefficient (cm2/sec):

4.8703E-10

Slope:

-3.1536E-04

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.05 - 0.003.9231E+02

R Square:

0.97499

Y-axis Intercept:

1.2372E-01

Wt. @ Zero	Wt. @ Zero	Adjustment to	Hardness				
Time, mg	Time, %	TGA Time, sec	Shore Scale				
Κ	L	M	N				
143.60	3.720	495	D				
TO 4 5							
TGA Dry	Equil. Wt. Of	Min. Wt. Used	Pre Exposure				
Wt., mg	Desorb. Sample	For FDC, mg.	Hardness				
138.450	138.41	138.410	72.6				
High Conc.	Low Conc.	Total Liquid	Post Desorption				
DC, cm2/sec	DC, cm2/sec	Sorbed, mg.	Hardness				
5.2834E-07	4.8703E-10	5.150					
0.1200 . 12 0 .	4.07 OOL-10	5.150	73.4				
R Square	R Square		Change in				
0.99973	0.97499		Hardness, %				
			1.1				
X Intercept	X Intercept		•••				
96.14	392.3		Mean Thickness,				
			cm.				
Y Intercept	Y Intercept		0.1579				
0.99862	0.12372						
			abs0115d				
Y Intercept	X Intercept	X Intercept	X Intercept				
as Wt., mg.	as Time, hrs.	as Time, min.	as Time, days				
143.402	42.8	256.5	1.8				
		May TO A VA	TO 4 14/4 A4 00				
TEMPERATURE STATIST	TICS	Max. TGA Wt.,	TGA Wt. At 20				
	1103	(orig data file) 142.407	min. Adjustment 141.729				
Column1		172.707	141.725				
		Sorption @ 7-12	Sorption @ 20				
Mean	30.00018454	min., mg	min., mg				
Standard Error	0.000465527	3.96	3.28				
Median	30						
Mode	29.999	Sorption @ 7-12	Sorption @ 20				
Standard Deviation	0.026895999	min., %	min., %				
Sample Variance	0.000723395	2.86	2.37				
Kurtosis	453.545885						
Skewness	-12.8162299	Rel. Error (%) of	Rel. Error (%) of				
Range	1.175	7-12 min. Sorp.	20 min. Sorp				
Minimum	29.141	-23.2	-36.3				
Maximum	30.316						
Sum	100140.616						
Count	3338						
Confidence Level(95.0%)	0.000912746						

Column1	
Mean	10.49
Standard Error	0.06770032
Median	10.495
Mode	#N/A
Standard Deviation	0.13540064
Sample Variance	0.01833333
Kurtosis	1.28419835
Skewness	-0.2175362
Range	0.33
Minimum	10.32
Maximum	10.65
Sum	41.96
Count	4
Confidence Level(95.0%)	0.21545284

ND/value ND/value A/D

### **THICKNESS STATISTICS**

	Column1						
	Mean	62.175					
	Standard Error	1.24121916					
	Median	61.7					
	Mode	#N/A					
	Standard Deviation	2.48243832					
	Sample Variance	6.1625					
	Kurtosis	1.90364577					
Unknown	Skewness	1.06966414					
abs0115d	Range	5.9					
	Minimum	59.7					
	Maximum	65.6					
	Sum	248.7					
	Count	4					
	Confidence Level(95.0%)	3.95011703					

abs0115d

### **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.999866631								
R Square	0.999733281								
Adjusted R Square	0.999723022								
Standard Error	0.001187972								
Observations	28								

# ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.137535524	0.137535524	97454.6928	5.35851E-48
Residual	26	3.66932E-05	1.41128E-06		
Total	27	0.137572217			

		Standard Error		P-value	Lower 95%	Upper 95%
Intercept	0.998619841	0.000977457				
X Variable 1	-0.010387099	3.32731E-05	-312.1773418	5.3585E-48	-0.010455493	-0.010318705

# LOW CONCENTRATION DIFFUSION COEFFICIENT

### **SUMMARY OUTPUT**

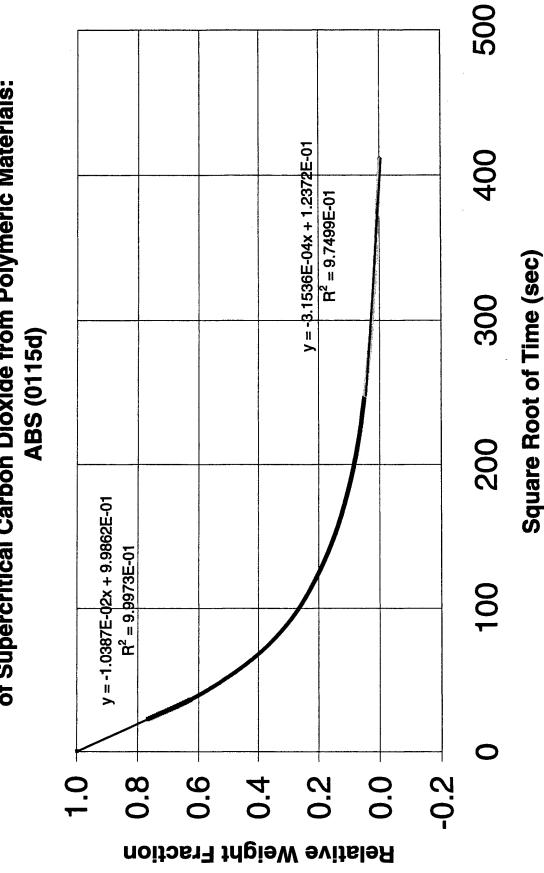
Regression Statistics									
Multiple R	0.987415446								
R Square	0.974989262								
Adjusted R Square	0.974970315								
Standard Error	0.002339443								
Observations	1322								

	df	SS	MS	F	Significance F
Regression	1	0.281625661	0.281625661	51457.3321	0
Residual	1320	0.007224352	5.47299E-06		
Total	1321	0.288850013			

		Standard Error		P-value	Lower 95%	Upper 95%
Intercept	0.123720738	0.000423464	292.1633424	0	0.122890002	0.124551474
X Variable 1	-0.000315365	1.39024E-06	-226.8420863	0	-0.000318092	-0.000312638

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:

*



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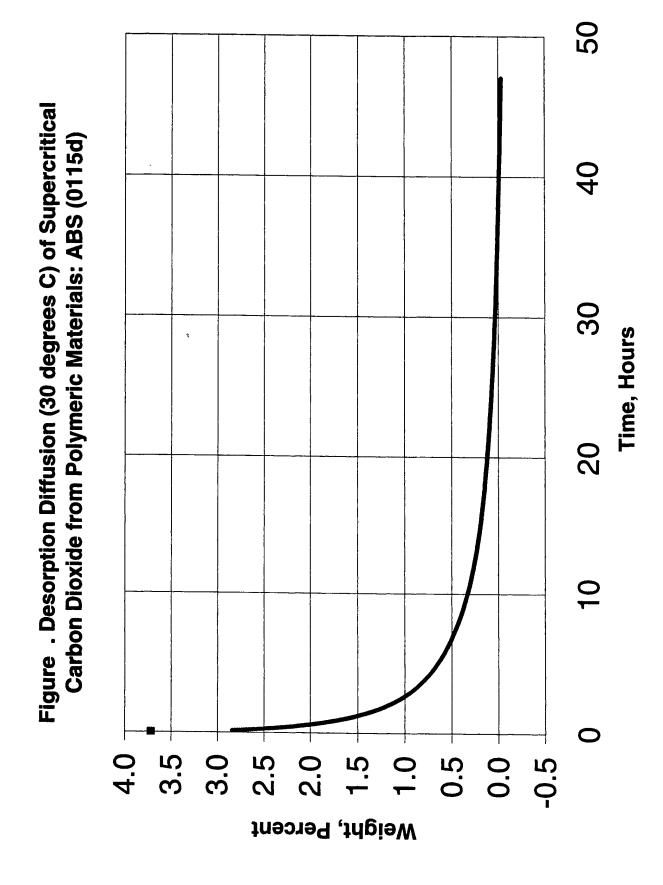


Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: ABS (0115d) 3.5 3.0 2.5 1.5 0.5 0.0 -0.5 2.0 1.0

50

40

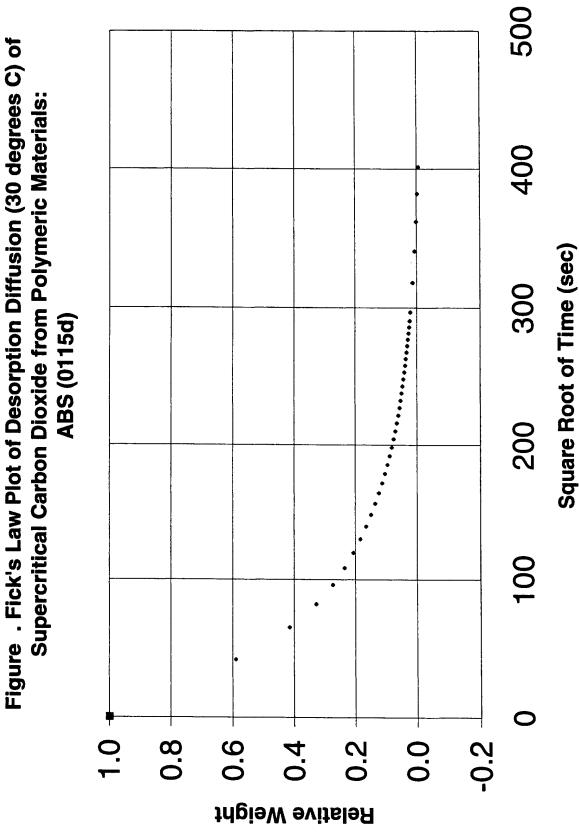
30

Time, Hours

133

Weight, Percent

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



Hardness Scale	Shore D	Hardness	72.6	:	Post Desorption	Hardness 73.4	t o	Change in	Hardness, %	1.10														1 400 E0 ET. 0.4E	1-1-30:30 E1. 0.13												
Adjustment to TGA Time, sec M	495			n=4																				477 007 04	Comment Disk: .4x0.06in., HP & 1400x50, Diy: 1G-136.447 M-136.36 E1. 6.13												
TGA Dry Wt., mg L	138.447	of Data	nmns	ginning with	***************************************							6			bs0115d.01	¥							ermai		P @ 1400x50												
ΧT	3.722	Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4		e 3	( <u>0</u> 0)	Ēu)	Hn = (((Cn - L4) / L4) * 100)	n = ((Cn - L4) / (J4 - L4))	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8) /N8) * 100)			Source binary TA data file : abs0115d.01	14 lines in the parameter block	of data		!	Module TGA Aluminum Pans	Sample ABS SCCO2 0115D	mg	Method DESORPTION-Isothermal	: :	SK: .4XO.UGIN., H	11	mu)		(mg)	1	8						
Wt. @ Zero Time, mg	143.60	Formulae Us	Uppercase le	Lowercase n		Dn = (An * 60) En = (Dn · 144	En = (Dn + M4) Fn = (Fn / 3600)	Gn = SQRT (En)	Hn = (((Cn -	In = ((Cn - L4	K4 = (((J4 - L	N16 = (((N12			Source binar	14 lines in th	3 channels of data	*******	Run 32	Module TGA	Sample ABS	Size 142.407 mg	Method DES	Operator BSI	Comment Di	NSIG 6	Sign Temperatur	Sigz Teimperature (BC)	Sig3 Weignt (mg)	Kcell 1.0000	Date 24-Apr-00	Time 15:21					
Fractional Wt. Loss	0.589	0.329	0.236	0.207	0.183	0.164	0.135	0.123	0.112	0.103	0.095	0.088	0.081	0.075	0.070	0.065	0.060	0.056	0.053	0.049	0.046	0.043	0.040	0.038	0.035	0.033	0.030	0.029	0.027	0.025	0.023	0.021	0.013	0.007	0.002	-0.003	-0.006
Wt. Loss, % H	2.194 1.544	1.021	0.878	0.769	0.683	0.612	0.501	0.457	0.417	0.384	0.353	0.326	0.301	0.280	0.259	0.241	0.225	0.209	0.196	0.183	0.171	0.160	0.149	0.140	0.131	באנים	0.13	0.0	0.099	0.092	0.085	0.077	0.049	0.025	0.007	0.00	-0.023
SQRT Adj. Wt. Loss, Time, sec % G H	65.12	96.15	108.38	119.36	129.42	138.75	155 75	163.60	171.08	178.24	185.13	191.76	198.18	204.39	210.43	216.29	222.01	227.57	233.00	238.31	243.51	248.59	253.58	258.47	263.26	267.97	272.60	2//.16	281.64	286.05	290.39	296.71	317.93	340.71	362.05	382.21	401.35
Adjusted Time, hr	0.483	1.8/3 2.568	3.263	3.958	4.653	5.348	6 739	7.434	8.130	8.825	9.520	10.215	10.910	11.605	12.300	12.995	13.691	14.386	15.081	15.776	16.471	17.166	17.862	18.557	19.252	19.947	20.642	21.338	22.034	22.729	23.424	24.455	28.078	32.245	36.411	40.578	44.745
Adjusted Time, sec	4240.560	6/42.620 9243.900	11746.200	14247.720	16750.020	19251.720	24259 440	26764.080	29269.560	31771.080	34272.480	36773.400	39275.280	41777.220	44279.760	46782.900	49287.660	51787.980	54290.400	56792.940	59297.040	61799.280	64302.540	66804.720	69307.800	71809.260	74311.740	76817.160	79320.960	81824.220	84326.520	88039.380	101080.620	116080.860	131081.160		161081.280
TGA Time, sec D	3745.560	6247.620 8748.900	11251.200	13752.720	16255.020	18756.720	23764 440	26269.080	28774.560	31276.080	33777.480	36278.400	38780.280	41282.220	43784.760	46287.900	48792.660	51292.980	53795.400	56297.940	58802.040	61304.280	63807.540	66309.720	68812.800	71314.260	73816.740	76322.160	78825.960	81329.220	83831.520	87544.380	100585.620	115585.860	130586.160	145586.220	160586.280
TGA Wt. mg C	141.484	139.861	139.662	139.512	139.392	139.294	139.212	139 080	139.025	138.978	138.936	138.898	138.864	138.834	138.806	138.781	138.758	138.737	138.718	138.701	138.684	138.668	138.653	138.641	138.628	138.615	138.604	138.594	138.584	138.574	138.565	138.553	138.515	138.482	138.456	138.434	138.415
Temp., deg C B	30.001	30.000 30.001	30.000	30.000	30.000	30.000	30.000	30.00	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000
TGA Time, min A	20.750	104.127 145.815	187.520	229.212	270.917	312.612	354.315	437.818	479.576	521.268	562.958	604.640	646.338	688.037	729.746	771.465	813.211	854.883	896.590	938.299	980.034	1021.738	1063.459	1105.162	1146.880	1188.571	1230.279	1272.036	1313.766	1355.487	1397.192	1459.073	1676.427	1926.431	2176.436	2426.437	2676.438

Blank

**Supercritical Carbon Dioxide** 

and

**Polyurethane** 

PU

### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

06-1-1

TGA Filename:

pu0124d

Property Test Specification: ASTM D471/D543 Material Name:

Polyurethane

Material Code:

PU

Material Supplier:

Unknown

Material Class:

U

Monomer Source:

Unknown

Base Polymer (Lot. No.): Unknown

Exposure Liquid:

Replicate Number:

Supercritical Carbon Dioxide

### **Pre-Exposure Data**

Measured Diameter (mm): Mean Diameter:

9.82

9.68

9.84 9.63

0.165

Measured Thickness (mils):

42.20

42.10

95% Confidence Level (CL): 42.00 42.10 95% Confidence Level (CL):

Mean Thickness: Initial Sample Weight (mg):

42.100

132.580

0.130

Initial Durometer Hardness:

96.9

00:15:00

Scale: SHORE

Α

### Sample Exposure Data

**Exposure Date:** 

05/03/00

Time:

10:26:00 10:41:00

Removal Date:

05/03/00

Time:

141.60

Total Exposure Time (h:m:s):

Weight of Sample After Exposure (mg):

Percent Weight Gain:

Weight Gain (mg):

9.02

<u>6.8</u>0

Final Durometer Hardness After Desorption:

97.0

Percent Hardness Change:

0.1

#### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.000

95% CL:

0.0009

Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

Time Lapse Between Sample Exposure and Sample Desorption (sec):

<u>555</u>

132.210

Sample Extracted (mg):

0.37

Solubility Corrected for Extractables (mg):

9.39

Sample Extracted (%):

0.28

Solubility Corrected for Extractables (%):

7.08

# Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm2/sec): Range Including 0,1 Intercept, Relative Weight Fraction:

2.5629E-07 1.00 - 0.62

-1.0684E-02

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.75 - 0.62

Slope: R Square:

0.99971

Y-axis Intercept:

9.3874E+01 1.0030E+00

Low Concentration Diffusion Coefficient (cm2/sec):

7.5549E-11

Slope:

-1.8344E-04

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.03 - 0.00 2.7272E+02

R Square:

0.80890

Y-axis Intercept:

5.0026E-02

Wt. @ Zero	Wt. @ Zero	Adjustment to	Hardness
Time, mg	Time, %	TGA Time, sec	Shore Scale
K	L	M	N
141.60	6.803	555	A
TGA Dry	Equil. Wt. Of	Min. Wt. Used	Pre Exposure
Wt., mg	Desorb. Sample	For FDC, mg.	Hardness
132.580	132.21	132.210	96.9
High Conc.	Low Conc.	Total Liquid	Post Desorption
DC, cm2/sec	DC, cm2/sec	Sorbed, mg.	Hardness
2.5629E-07	7.5549E-11	9.020	97.0
R Square 0.99971	R Square 0.80890		Change in Hardness, % 0.1
X Intercept	X Intercept		Mean Thickness,
93.87	272.7		cm.
Y Intercept	Y Intercept		0.1069
1.00296  Y Intercept as Wt., mg. 142.019	0.05003  X Intercept as Time, hrs.	X Intercept as Time, min.	pu0124d X Intercept as Time, days
TEMPERATURE STATIS	20.7 TICS	124.0 Max. TGA Wt., (orig data file) 139.304	0.9 TGA Wt. At 20 min. Adjustment 138.139
Column1			
Mean	30.00004864	Sorption @ 7-12	Sorption @ 20
Standard Error	0.000470253	min., mg	min., mg
Median	30	6.72	5.56
Mode Standard Deviation Sample Variance Kurtosis		Sorption @ 7-12 min., % 5.07	Sorption @ 20 min., % 4.19
Skewness	-15.26590206	Rel. Error (%) of	Rel. Error (%) of
Range	1.06	7-12 min. Sorp.	20 min. Sorp
Minimum	29.197	-25.5	-38.4
Maximum Sum Count Confidence Level(95.0%)	30.257 86970.141 2899 0.000922064		

### **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.999852658								
R Square	0.999705338								
Adjusted R Square	0.99969306								
Standard Error	0.001298315								
Observations	26								

### ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.137252202	0.137252202	81425.24	6.90655E-44
Residual	24	4.04549E-05	1.68562E-06		
Total	25	0.137292657			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	1.002957462	0.001116626	898.2035895	7.7393E-56	1.00065286	1.005262064
X Variable 1	-0.010684114	3.7442E-05	-285.3510819	6.9066E-44	-0.01076139	-0.010606837

### LOW CONCENTRATION DIFFUSION COEFFICIENT

### **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.8993903								
R Square	0.808902912								
Adjusted R Square	0.808806203								
Standard Error	0.003258879								
Observations	1978								

	df	SS	MS	F	Significance F
Regression	1	0.088831255	0.088831255	8364.29361	0
Residual	1976	0.0209857	1.06203E-05		
Total	1977	0.109816955			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%_
Intercept	0.050025635	0.000482698	103.637583	0	0.049078985	0.050972286
X Variable 1	-0.000183435	2.00571E-06	-91.4565121	0	-0.000187369	-0.000179502

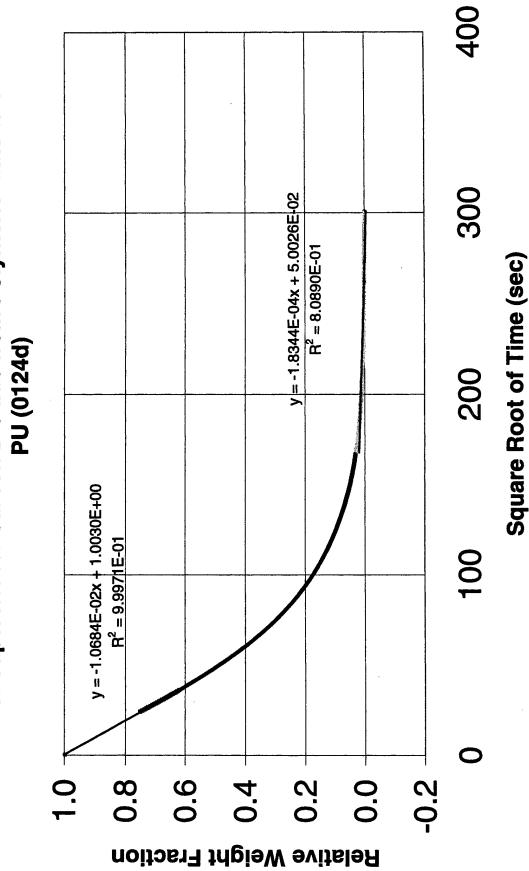
Column1	
Mean	9.7425
Standard Error	0.05170026
Median	9.75
Mode	#N/A
Standard Deviation	0.10340052
Sample Variance	0.01069167
Kurtosis	-4.6970753
Skewness	-0.1662108
Range	0.21
Minimum	9.63
Maximum	9.84
Sum	38.97
Count	4
Confidence Level(95.0%)	0.16453345

# THICKNESS STATISTICS

	Column1				
	Mean	42.1			
	Standard Error	0.04082483			
	Median	42.1			
	Mode	42.1			
	Standard Deviation	0.08164966			
	Sample Variance	0.00666667			
	Kurtosis	1.5			
Unknown	Skewness	0			
pu0124d	Range	0.2			
	Minimum	42			
	Maximum	42.2			
	Sum	168.4			
	Count	4			
	Confidence Level(95.0%)	0.12992295			

ND/value ND/value A/D

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



APPENDIX B

Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PU (0124d) 7.0 6.0 1.0 2.0 0.0

30

25

20

S

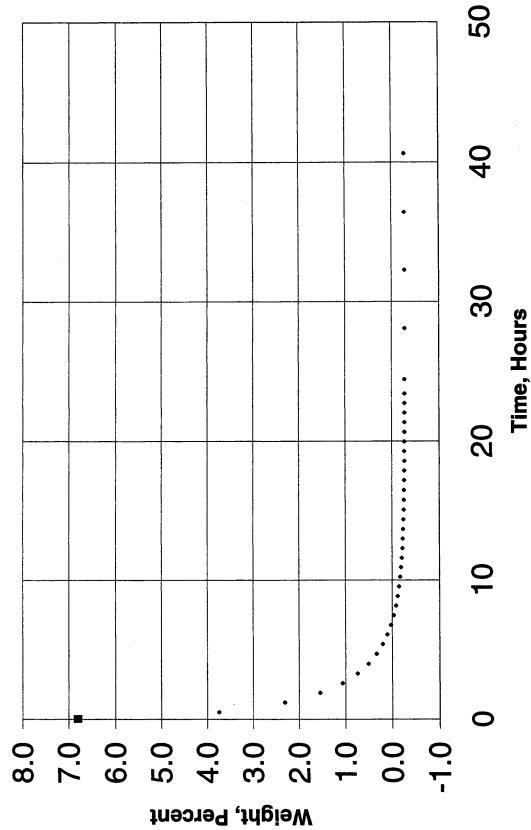
-1.0

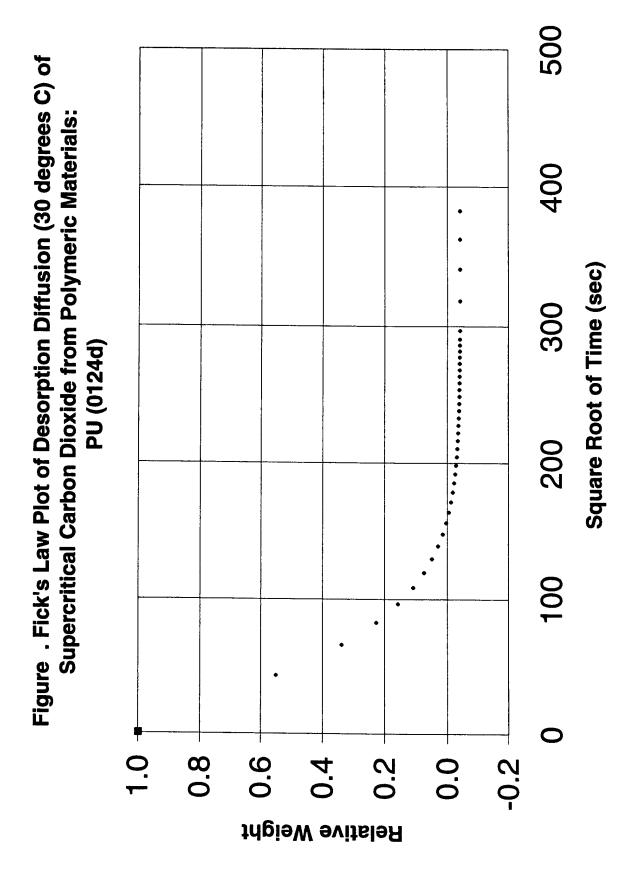
Time, Hours

142

Weight, Percent

Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PU (0124d)





Hardness	Scale	Z	Shore A		Pre Exposure	Hardness	96.9		Post Desoration	Hardness	97.0	!	Change in	Hardness, %	0.10															-132.81 ET:9:15				-							
Adjustment to	TGA Time, sec	Σ	555					4																						Comment Disk: .382x.0668IN, HP @ 1400x50, Dry: TG-132.575 M-132.81 ET:9:15											
TGA Dry	Wt., mg	ئ	132.575			f Data	Imns	ainnina with n									_			10124d.01	*							rmal		HP @ 1400x5											
Wt. @ Zero	Time, %	¥	6.807			for Analysis of	rs refer to colu	fers to row. be				_	. =	/ L4) * 100)	(74 - [4))	/ L4) * 100)	N8) /N8) * 100			A data file: pu	parameter bloc	data			uminum Pans	CO2 124D	5	PTION-Isothe		.382x.0668IN,		_	ire (øC)	6							
Wt. @ Zero	Time, mg	٠,	141.60			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row. beginning with n=4		Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4)/L4) * 100)	n = ((Cn - L4) / (J4 - L4))	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8)/N8) * 100)	•		Source binary TA data file: pu0124d.01	14 lines in the parameter block	3 channels of data		Run 34	Module TGA Aluminum Pans	Sample PU SCCO2 124D	Size 139.304 mg	Method DESORPTION-Isothermal	Operator WJS	Somment Disk:	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 3-May-00	Time 10:51				
Fractional	Wt. Loss	- }	0.551	0.340	0.228	0.157	0.109		0.049	_		_		_	-0.017	-0.021	-0.025	-0.028	-0.030	-0.032	-0.033	-0.034	-0.035	-0.036	_		-0.038	_	_	_				-0.040	-0.040	-0.040	0.040	-0.040	0.040	0.040	0.040
Wt. Loss,	%:	ב i	3.748	2.311	1.551	1.070	0.741	0.508	0.336	0.205	0.105	0.028	-0.031	-0.078	-0.115	-0.146	-0.169	-0.188	-0.204	-0.216	-0.226	-0.235	-0.241	-0.247	-0.253	-0.256	-0.260	-0.262	-0.265	-0.266	-0.267	-0.269	-0.270	-0.271	-0.272	-0.272	-0.274	-0.275	-0.275	-0.275	-0.275
SORT Adj. Wt. Loss,	lime, sec	5 (	42.44	65.60	82.49	96.47	108.67	119.62	129.66	138.97	147.70	155.94	163.77	171.24	178.40	185.28	191.92	198.32	204.53	210.56	216.42	222.13	227.70	233.13	238.44	243.63	248.71	253.69	258.58	263.38	268.09	272.71	277.26	281.74	286.15	290.50	296.85	318.10	340.87	362.20	382.35
Adjusted		L	0.500	1.195	1.890	2.585	3.280	3.975	4.670	5.365	6.060	6.755	7.450	8.146	8.841	9.536	10.231	10.926	11.620	12.315	13.010	13.706	14.401	15.097	15.793	16.488	17.183	17.878	18.573	19.268	19.964	20.659	21.354	22.050	22.746	23.441	24.478	28.108	32.275	36.442	40.609
Adjusted	ime, sec	u ;	1801.020	4302.720	6805.140	9306.120	11808.120	14309.640	16810.980	19313.940	21815.460	24318.600	26820.060	29323.860	31826.100	34330.440	36831.960	39332.160	41832.960	44335.140	46837.500	49342.860	51845.280	54349.020	56853.300	59355.960	61857.660	64360.920	66863.580	69366.540	71869.920	74371.440	76875.300	79378.560	81884.280	84387.420	88122.060	101190.000	116191.320	131191.740	146192.040
TGA Time,	S (	3	1246.020	3747.720	6250.140	8751.120	11253.120	13754.640	16255.980	18758.940	21260.460	23763.600	26265.060	28768.860	31271.100	33775.440	36276.960	38777.160	41277.960	43780.140	46282.500	48787.860	51290.280	53794.020	56298.300	58800.960	61302.660	63805.920	66308.580	68811.540	71314.920	73816.440	76320.300	78823.560	81329,280	83832.420	87567.060	100635.000	115636.320	130636.740	145637.040
TGA	Wt., mg	ٔ د	137.544	135.639	134,631	133.993	133.558	133.248	133.020	132.847	132.714	132.612	132.534	132.472	132.423	132.382	132.351	132.326	132.305	132.288	132.275	132.264	132.256	132.247	132.240	132,235	132.230	132.227	132.224	132.222	132.221	132.219	132.217	132.216	132.215	132.214	132.212	132.210	132.210	132.210	132.211
Temp.	ပ ရွှင်	מ	29.997	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.00	30.000
TGA Time,	Ę.	∢ ;	20.767	62.462	104.169	145.852	187.552	229.244	270.933	312.649	354.341	396.060	437.751	479.481	521.185	562.924	604.616	646.286	687.966	729.669	771.375	813.131	854.838	896.567	938.305	980.016	1021.711	1063.432	1105.143	1146.859	1188.582	1230.274	1272.005	1313.726	1355.488	1397.207	1459.451	1677.250	1927.272	2177.279	2427.284

**Supercritical Carbon Dioxide** 

and

Silicone Modified Organic

SMO

### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

07-1-1

TGA Filename:

smo0129d

Material Name:

Property Test Specification: ASTM D471/D543 Silicone Modified Organic

Material Code:

SMO

Material Supplier:

Unknown

**Material Class:** 

Q

Monomer Source:

Unknown

Base Polymer (Lot. No.): Unknown

**Exposure Liquid:** 

Supercritical Carbon Dioxide

Replicate Number:

1

**Pre-Exposure Data** 

Measured Diameter (mm):

9.83

**75.00** 

9.91

9.80 95% Confidence Level (CL):

0.085

Mean Diameter: Measured Thickness (mils):

9.865

74.90

75.00 75.10

Mean Thickness:

75.000

148.460

9.90

95% Confidence Level (CL): 0.130

Initial Sample Weight (mg): Initial Durometer Hardness:

58.6

Scale: SHORE

Α

### Sample Exposure Data

**Exposure Date:** 

05/08/00

Time:

14:25:00

Removal Date:

05/08/00

Time: 14:40:00

Total Exposure Time (h:m:s):

7.34

00:15:00

Weight of Sample After Exposure (mg):

<u>155.80</u>

Weight Gain (mg):

Percent Weight Gain:

4.94

Final Durometer Hardness After Desorption:

58.9

Percent Hardness Change:

0.5

#### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.000

95% CL:

0.0013

Time Lapse Between Sample Exposure and Sample Desorption (sec): Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

615

148.750

Sample Extracted (mg):

-0.29

Solubility Corrected for Extractables (mg):

<u>7.34</u>

Sample Extracted (%):

-0.20

Solubility Corrected for Extractables (%):

4.94

## **Diffusion Coefficient Data: Fick's Law**

High Concentration Diffusion Coefficient (cm2/sec): Range Including 0,1 Intercept, Relative Weight Fraction: 5.1028E-07

Linear Range of Desorption, Relative Weight Fraction:

X-axis Intercept:

1.00 - 0.660.79 - 0.66

Slope: -8.4624E-03 R Square:

0.99995

Y-axis Intercept:

1.1827E+02 1.0008E+00

Low Concentration Diffusion Coefficient (cm2/sec):

2.8522E-09 0.07 - 0.04

Slope: R Square:

-6.3267E-04 0.99463

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept: Y-axis Intercept:

3.3044E+02 2.0906E-01

Wt. @ Zero	Wt. @ Zero	Adjustment to	Hardness
Time, mg	Time, %	TGA Time, sec	Shore Scale
K	L	M	N
155.80	4.944	615	Α
TGA Dry	Equil. Wt. Of	Min. Wt. Used	Pre Exposure
Wt., mg	Desorb. Sample	For FDC, mg.	Hardness
148.460	148.75	148.460	58.6
High Conc.	Low Conc.	Total Liquid	Post Desorption
DC, cm2/sec	DC, cm2/sec	Sorbed, mg.	Hardness
5.1028E-07	2.8522E-09	7.340	58.9
R Square	R Square		Change in
0.9995	0.99463		Hardness, %
			0.5
X Intercept	X Intercept		
118.27	330.4		Mean Thickness,
Y Intercept	Y Intercept		cm. 0.1905
1.00083	0.20906		0.1905
	0.2000		smo0129d
Y Intercept	X Intercept	X Intercept	X Intercept
as Wt., mg.	as Time, hrs.	as Time, min.	as Time, days
155.930	30.3	182.0	1.3
		Max. TGA Wt.,	TGA Wt. At 20
TEMPERATURE STATIS	TICS	(orig data file)	min. Adjustment
		154.274	153.654
Column1		'	
Mean	30.00005822	Sorption @ 7-12	Sorption @ 20
Standard Error	0.000677744	min., mg 5.81	min., mg
Median	30	5.61	5.19
Mode	30	Sorption @ 7-12	Sorption @ 20
Standard Deviation	0.033354348	min., %	min., %
Sample Variance	0.001112513	3.92	3.50
Kurtosis	358.3810313	0.02	5.50
Skewness	-12.57316686	Rel. Error (%) of	Rel. Error (%) of
Range	1.248	7-12 min. Sorp.	20 min. Sorp
Minimum	29.076	-20.8	-29.2
Maximum	30.324		20.2
Sum	72660.141		
Count	2422		
Confidence Level(95.0%)	0.001329017		

### HIGH CONCENTRATION DIFFUSION COEFFICIENT

### **SUMMARY OUTPUT**

Regression S	tatistics
Multiple R	0.999975055
R Square	0.99995011
Adjusted R Square	0.999948643
Standard Error	0.000437112
Observations	36

### ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.13020571	0.13020571	681465.356	9.98267E-75
Residual	34	6.49629E-06	1.91067E-07		
Total	35	0.130212206			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	1.00083354	0.000340523	2939.109506	1.7733E-93	1.000141515	1.001525565
X Variable 1	-0.008462427	1.02512E-05	-825.5091498	9.9827E-75	-0.00848326	-0.008441594

### LOW CONCENTRATION DIFFUSION COEFFICIENT

### **SUMMARY OUTPUT**

Regression Statistics										
Multiple R	0.997312275									
R Square	0.994631773									
Adjusted R Square	0.994625129									
Standard Error	0.000662618									
Observations	810									

### **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.06573076	0.06573076	149707.254	0
Residual	808	0.000354762	4.39062E-07		
Total	809	0.066085522			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.20905919	0.000404425	516.9300144	0	0.208265344	0.209853037
X Variable 1	-0.000632674	1.63515E-06	-386.9202161	0	-0.000635884	-0.000629464

# **DIAMETER STATISTICS**

Column1	
Mean	9.86
Standard Error	0.02677063
Median	9.865
Mode	#N/A
Standard Deviation	0.05354126
Sample Variance	0.00286667
Kurtosis	-4.3409951
Skewness	-0.2345507
Range	0.11
Minimum	9.8
Maximum	9.91
Sum	39.44
Count	4
Confidence Level(95.0%)	0.08519617

# **THICKNESS STATISTICS**

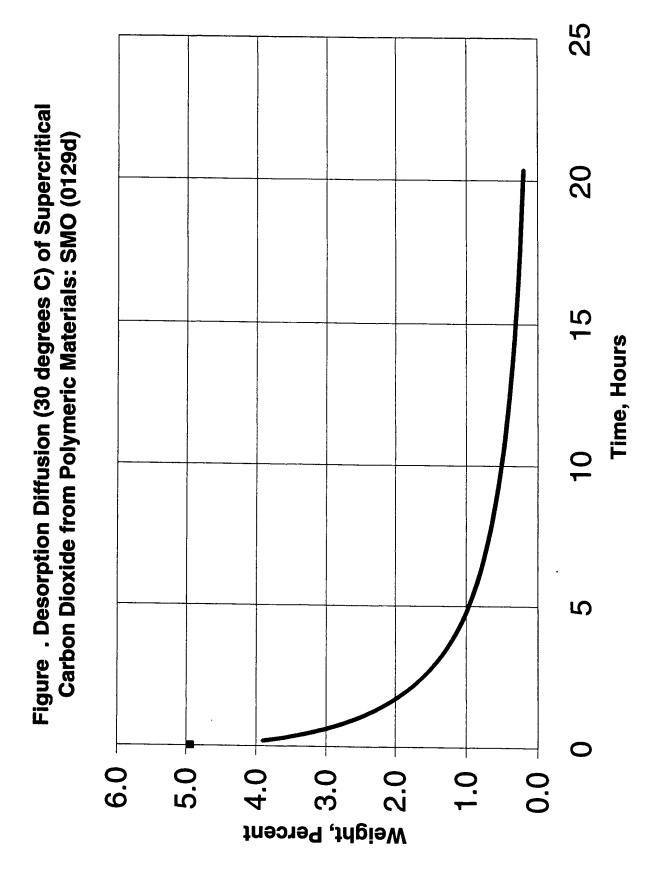
	Column1	
	Mean	75
	Standard Error	0.04082483
	Median	75
	Mode	75
	Standard Deviation	0.08164966
	Sample Variance	0.00666667
	Kurtosis	1.5
Unknown	Skewness	0
smo0129d	Range	0.2
	Minimum	74.9
	Maximum	<i>7</i> 5.1
	Sum	300
	Count	4
	Confidence Level(95.0%)	0.12992295

ND/value ND/value A/D

400 Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: = -6.3267E - 04x + 2.0906E - 01300  $R^2 = 9.9463 = -01$ SMO (0129d) 200 V = -8.4624E - 03x + 1.0008E + 00 $R^2 = 9.9995E-01$ 100 0.5 9.0 0.3 0.8 0.4

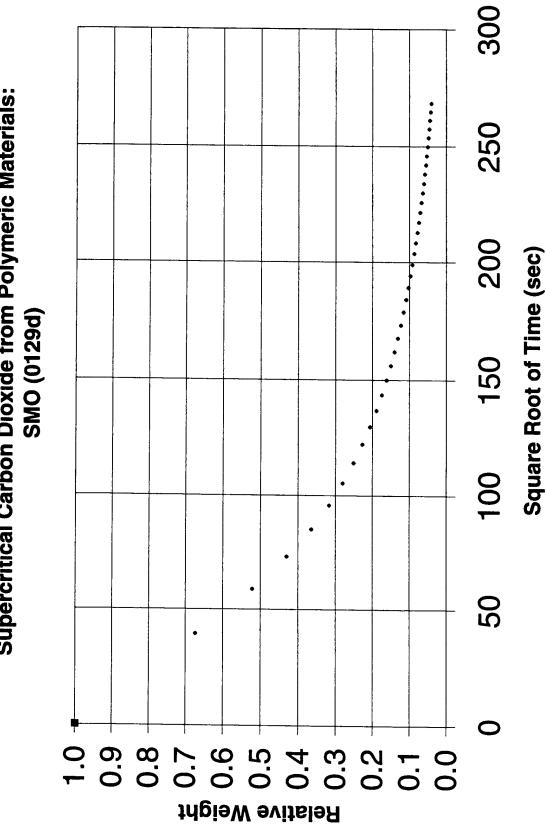
Square Root of Time (sec)

Relative Weight Fraction



25 Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: SMO (0129d) 20 Time, Hours S 5.0 4.0 2.0 1.0 6.0 3.0 Weight, Percent

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



Hardness Scale N	Shore A	Ĺ	Fre Exposure	Hardness	58.6		Post Desorption	Hardness	58.9		Change in	Hardness, %	0.51														:	148.461 ET:10:15			-							
Adjustment to TGA Time, sec M	615					4																						Comment .38"x.075",HP @ 1400x50,Dry:DS-148.3 M-148.54 TG-148.461 ET:10:15										•
TGA Dry Wt., mg L	148.461		ļ	of Data	nmns	ginning with n									6			mo0129d.01	¥					129D Disk		ermal		.00x50,Dry:DS										
Wt. @ Zero Time, % K	4.943			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4			<u>.</u>	6	ĵ.	4)/L4)*100)	/ (74 - L4))	1) / L4) * 100)	N16 = (((N12 - N8)/N8) * 100)			Source binary TA data file: smo0129d.01	14 lines in the parameter block	f data			Module TGA Aluminum Pans	Sample SMO GPM SCCO2 0129D Disk	gn gn	Method DESORPTION-Isothermal		x.075",HP@14		Ê	ture (øC)	ng)		0				
Wt. @ Zero Time, mg	155.80			Formulae Use	Uppercase lett	Lowercase n r		Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	tn = ((Cn - L4) / (J4 - L4))	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 -			Source binary	14 lines in the	3 channels of data		Run 36	Module TGA A	Sample SMO	Size 154.274 mg	Method DESO	Operator BSI	Comment .38"	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 8-May-00	Time 14:51			
Fractional Wt. Loss	0.674	0.522	0.430	0.365	0.318	0.280	0.251	0.227	0.207	0.189	0.175	0.162	0.150	0.140	0.131	0.123	0.116	0.109	0.103	0.097	0.092	0.087	0.082	0.078	0.074	0.00	0.067	0.064	0.061	0.058	0.055	0.053	0.050	0.048	0.046	0.044	0.042	0.040
Wt. Loss, H	3.329	2.580	2.125	1.807	1.570	1.386	1.239	1.121	1.021	0.936	0.864	0.800	0.744	0.694	0.649	0.610	0.573	0.539	0.509	0.480	0.454	0.429	0.407	0.385	0.366	0.348	0.331	0.315	0.300	0.286	0.272	0.260	0.248	0.237	0.226	0.217	0.207	0.199
SQRT Adj. Wt. Loss, Time, sec % G H	39.50	58.82	73.22	85.23	95.75	105.21	113.91	121.97	129.54	136.68	143.47	149.95	156.16	162.13	167.89	173.46	178.86	184.10	189.19	194.15	198.99	203.71	208.32	212.84	217.26	221.60	225.85	230.05	234.11	238.14	242.10	245.99	249.83	253.60	257.32	261.00	264.61	268.18
Adjusted Time, hr F	0.433	0.961	1.489	2.018	2.547	3.075	3.604	4.133	4.661	5.189	5.717	6.245	6.774	7.302	7.830	8.358	8.887	9.415	9.943	10.471	10.999	11.527	12.055	12.583	13.112	13.640	14.169	14.696	15.224	15.753	16.281	16.809	17.337	17.865	18.393	18.922	19.450	19.978
Adjusted Time, sec E	1559.940	3460.200	5360.820	7263.420	9167.580	11070.180	12975.720	14877.300	16779.840	18681.060	20582.580	22483,680	24386.160	26286.960	28187.700	30088.680	31991.640	33892.620	35793.060	37695.720	39596.940	41497.980	43398.420	45300.420	47203.680	49105.020	51006.660	52907.040	54808.020	56709.120	58610.340	60511.320	62412.600	64313.520	66215.280	68119.020	70019.940	71921.340
TGA Time, sec D	944.940	2845.200	4745.820	6648.420	8552.580	10455.180	12360.720	14262.300	16164.840	18066.060	19967.580	21868.680	23771.160	25671.960	27572.700	29473.680	31376.640	33277.620	35178.060	37080.720	38981.940	40882.980	42783.420	44685.420	46588.680	48490.020	50391.660	52292.040	54193.020	56094.120	57995.340	59896.320	61797.600	63698.520	65600.280	67504.020	69404.940	71306.340
TGA Wt., mg C	153.404	152.291	151.616	151.143	150.792	150.519	150.301	150.125	149.977	149.851	149.743	149.648	149.565	149.492	149.425	149.366	149.312	149.261	149.216	149.173	149.135	149.098	149.065	149.033	149.004	148.977	148.952	148.928	148.906	148.885	148.865	148.847	148.829	148.813	148.797	148.783	148.769	148.756
Temp., deg C B	29.995	30.000	30.000	30.00	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	29.999	30.001	30.000	30.000	30.000
TGA Time, min A	15.749	47.420	79.097	110.807	142.543	174.253	206.012	237.705	269.414	301.101	332.793	364.478	396.186	427.866	459.545	491.228	522.944	554.627	586.301	618.012	649.699	681.383	713.057	744.757	776.478	808.167	839.861	871.534	903.217	934.902	966.589	998.272	1029.960	1061.642	1093.338	1125.067	1156.749	1188.439

**Supercritical Carbon Dioxide** and

Polyethylene-propylene

PEP

### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

08-1-1

TGA Filename:

pep0130d

Material Name:

Property Test Specification: ASTM D471/D543

Material Code:

PEP

Material Supplier:

Polyethylene-propylene **Smithers Scientific** 

Material Class:

**EPM** 

Monomer Source:

Dupont

Base Polymer (Lot. No.): Nordel 2744

**Exposure Liquid:** 

Supercritical Carbon Dioxide

Replicate Number:

1

**Pre-Exposure Data** 

Measured Diameter (mm):

9.82

9.87

9.81 9.87 95% Confidence Level (CL):

0.051

Mean Diameter: Measured Thickness (mils):

9.845 48.40

48.50

49.20

48.60

Mean Thickness:

48.675

95% Confidence Level (CL):

Initial Sample Weight (mg): **Initial Durometer Hardness:**  114.050

93.6

Scale: SHORE

0.572

Sample Exposure Data

**Exposure Date:** 

05/09/00

Time:

11:21:00

Removal Date:

05/09/00

Time:

11:36:00

Total Exposure Time (h:m:s): Weight Gain (mg):

3.55

Weight of Sample After Exposure (mg): 00:15:00

Percent Weight Gain:

117.60 3.11

Percent Hardness Change:

Final Durometer Hardness After Desorption:

93.9

Α

0.3

### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C): Time Lapse Between Sample Exposure and Sample Desorption (sec):

30.000

95% CL:

0.0015

Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

Solubility Corrected for Extractables (mg):

635 113.930

Sample Extracted (mg):

0.12 0.11

Solubility Corrected for Extractables (%):

3.67

Sample Extracted (%):

3.22

#### Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm2/sec):

Range Including 0,1 Intercept, Relative Weight Fraction:

1.1302E-06 1.00 - 0.30

Linear Range of Desorption, Relative Weight Fraction: -1.9405E-02

X-axis Intercept:

Y-axis Intercept:

0.50 - 0.305.1234E+01

Slope: R Square: 0.99918

Y-axis Intercept:

9.9420E-01

Low Concentration Diffusion Coefficient (cm2/sec): Linear Range of Desorption, Relative Weight Fraction: 5.7250E-11

Slope:

R Square:

-1.3811E-04 0.96751

X-axis Intercept:

0.03 - 0.002.7238E+02 3.7619E-02

Time, mg	Wt. @ Zero	Wt. @ Zero	Adjustment to	Hardness
K 117.60         L 3.113         M 635         N A           TGA Dry Wt., mg 114.050         Equil. Wt. Of Desorb. Sample 113.93         Min. Wt. Used For FDC, mg. 113.930         Pre Exposure Hardness 93.6           High Conc. DC, cm2/sec 1.1302E-06         Low Conc. DC, cm2/sec DC, cm2/sec 1.1302E-06         Total Liquid Sorbed, mg. 3.550         Post Desorption Hardness 93.9           R Square 0.99918         N 0.96751         Change in Hardness, % 0.3           X Intercept 51.23         X Intercept 272.4         Mean Thickness, cm. 0.1236           Y Intercept as Wt., mg. 116.918         X Intercept as Time, hrs. 116.918         X Intercept as Time, hrs. 115.799         X Intercept as Time, days 0.9           TEMPERATURE STATISTICS         Max. TGA Wt. (orig data file) 115.799         Copption @ 7-12 min., mg 1.75         Sorption @ 20 min., mg 1.07           Mean Standard Error Mode         30.00029496 0.000777476         min., mg 1.75         Sorption @ 20 min., mg 1.07           Standard Deviation Scample Variance Wurdosis Skewness         0.040540732 0.001643551 1.53         Neel. Error (%) of 7-12 min. Sorp. 7-12 min. Sorp. 7-12 min. Sorp. 7-12 min. Sorp. 7-10.0         Rel. Error (%) of 20 min. Sorp           Maximum         28.823 -50.7         -50.7         -70.0	Time, mg	Time, %		
TGA Dry Equil. Wt. Of Win. Wt. Used Wt., mg Desorb. Sample 114.050 113.93 113.930 Post Desorption DC, cm2/sec 1.1302E-06 5.7250E-11 3.550 93.9  R Square R Square 0.99918 0.96751 Change in Hardness, % 0.3  X Intercept 51.23 272.4 Mean Thickness, cm. 0.1236	K	L		
Wt., mg 114.050         Desorb. Sample 113.93         For FDC, mg. 113.930         Hardness 93.6           High Conc. DC, cm2/sec 1.1302E-06         Low Conc. DC, cm2/sec 1.1302E-06         Total Liquid Sorbed, mg. 3.550         Post Desorption Hardness 93.9           R Square 0.99918         R Square 0.99751         Change in Hardness, % 0.3           X Intercept 51.23         X Intercept 272.4         Mean Thickness, cm. 0.1236           Y Intercept 0.99420         X Intercept as Time, hrs. 116.918         X Intercept as Time, hrs. 20.6         X Intercept as Time, min. 123.7         TGA Wt., (orig data file) 115.799         TGA Wt. At 20 min. Adjustment 115.116           TEMPERATURE STATISTICS         Sorption @ 7-12 min., mg 1.07         Sorption @ 20 min., mg 1.07           Mean Standard Error         30.00029496 0.000777476         Sorption @ 7-12 min., mg 1.75         Sorption @ 20 min., mg 1.07           Standard Deviation Sample Variance Wurtosis 367.9197048 Skewness         0.001643551 1.59879076         1.53 Rel. Error (%) of 7-12 min. Sorp. 40.00         Rel. Error (%) of 7-12 min. Sorp. -70.0           Maximum         28.823 -50.7         -50.7         -70.0	117.60	3.113	635	
Wt., mg 114.050         Desorb. Sample 113.93         For FDC, mg. 113.930         Hardness 93.6           High Conc. DC, cm2/sec 1.1302E-06         Low Conc. DC, cm2/sec 1.1302E-06         Total Liquid Sorbed, mg. 3.550         Post Desorption Hardness 93.9           R Square 0.99918         R Square 0.99751         Change in Hardness, % 0.3           X Intercept 51.23         X Intercept 272.4         Mean Thickness, cm. 0.1236           Y Intercept 0.99420         X Intercept as Time, hrs. 116.918         X Intercept as Time, hrs. 20.6         X Intercept as Time, min. 123.7         TGA Wt., (orig data file) 115.799         TGA Wt. At 20 min. Adjustment 115.116           TEMPERATURE STATISTICS         Sorption @ 7-12 min., mg 1.07         Sorption @ 20 min., mg 1.07           Mean Standard Error         30.00029496 0.000777476         Sorption @ 7-12 min., mg 1.75         Sorption @ 20 min., mg 1.07           Standard Deviation Sample Variance Wurtosis 367.9197048 Skewness         0.001643551 1.59879076         1.53 Rel. Error (%) of 7-12 min. Sorp. 40.00         Rel. Error (%) of 7-12 min. Sorp. -70.0           Maximum         28.823 -50.7         -50.7         -70.0				
Wt., mg         Desorb. Sample         For FDC, mg.         Hardness           114.050         113.93         113.930         Post Desorption           High Conc.         Low Conc.         Total Liquid         Post Desorption           DC, cm2/sec         DC, cm2/sec         Sorbed, mg.         Hardness           1.1302E-06         5.7250E-11         3.550         Post Desorption           R Square         0.9918         0.96751         Change in           Number of the post o		Equil. Wt. Of	Min. Wt. Used	Pre Exposure
High Conc.		Desorb. Sample	For FDC, mg.	
DC, cm2/sec	114.050	113.93	113.930	93.6
DC, cm2/sec	High Oams			
1.1302E-06 5.7250E-11 3.550 93.9  R Square 0.99918 0.96751 Change in Hardness, % 0.3  X Intercept 51.23 272.4 Mean Thickness, cm. 0.1236  Y Intercept Y Intercept 0.99420 0.03762  Y Intercept as Wt., mg. as Time, hrs. 116.918 20.6 Max. TGA Wt., (orig data file) 115.799  TEMPERATURE STATISTICS  Mean 30.00029496 min., mg 1.75 1.07  Median 30 Mode 30 Sorption @ 7-12 min., mg 1.07  Median 30 Mode 30 Sorption @ 7-12 Sorption @ 20 min., mg 1.07  Median 30 Mode 30 Sorption @ 7-12 Sorption @ 20 Standard Deviation 0.040540732 min., % 1.53 0.93  Sample Variance 0.001643551 1.53 0.93  Kurtosis 367.9197048 Skewness -11.59879076 Rel. Error (%) of Range 1.577 7-12 min. Sorp. Minimum 28.823 -50.7 7-12 min. Sorp. Minimum 28.823 -50.7 -70.0				
R Square 0.99918 0.96751 Change in Hardness, % 0.3  X Intercept 51.23 272.4 Mean Thickness, cm. 0.1236  V Intercept V Intercept as Wt., mg. as Time, hrs. 116.918 20.6 TEMPERATURE STATISTICS  Mean 30.00029496 Standard Error Median 30 Mode 30 Standard Deviation Sample Variance Median Sample Variance Standard Deviation Sample Variance Standard Deviation Sample Variance Standard Seven Seven Seven Standard Seven Sev				Hardness
Near	1.1302E-06	5.7250E-11	3.550	93.9
Near   Standard Error   Column1   Column1   Column2   Column3   Column4   Column4   Column4   Column5   Column5   Column6   Column6   Column6   Column6   Column7   Column7   Column7   Column6   Column7   Column8	P Square	D C		
X Intercept 51.23 272.4 Mean Thickness, 50m. 0.1236  Y Intercept 0.99420 0.03762  Y Intercept as Wt., mg. as Time, hrs. 116.918 20.6 X Intercept as Time, min. 123.7 0.9  TEMPERATURE STATISTICS  Mean 30.00029496 min., mg				
X Intercept   51.23   272.4   Mean Thickness, cm.   0.1236     Y Intercept   Q.09420   0.03762	0.99916	0.96/51		
Y   Intercept   Y   Intercept   0.99420   0.03762	Y Intercent	V Intercent		0.3
Y   Intercept   0.99420   0.03762	•	•		
Variable	51.23	2/2.4		
0.99420         0.03762           Y Intercept as Wt., mg. 116.918         X Intercept as Time, hrs. 20.6         X Intercept as Time, min. 123.7         X Intercept as Time, days 0.9           TEMPERATURE STATISTICS         Max. TGA Wt., (orig data file) at file) 115.799         TGA Wt. At 20 min. Adjustment 115.116           Column1         Sorption @ 7-12 Sorption @ 20 min., mg 1.07           Mean Standard Error 0.000777476         0.000777476         1.75         1.07           Median 30 Mode 30 Sorption @ 7-12 Standard Deviation Sample Variance 0.040540732 min., % min., % 0.93         Sorption @ 20 min., % 0.93         Min., % 0.93           Kurtosis 367.9197048 Skewness -11.59879076 Range 1.577 7-12 min. Sorp. Minimum 28.823 -50.7 -50.7         Rel. Error (%) of 7-12 min. Sorp70.0         Rel. Error (%) of -70.0           Maximum 30.4         -50.7 -70.0         -70.0	Y Intercept	Y Intercent		
Y Intercept as Wt., mg. 116.918         X Intercept as Time, hrs. 20.6         X Intercept as Time, min. 123.7         X Intercept as Time, days 0.9           TEMPERATURE STATISTICS         Max. TGA Wt., (orig data file) as Time, days 0.9           Column1         Sorption @ 7-12 min. Adjustment 115.799           TITEMPERATURE STATISTICS         Sorption @ 7-12 min., Adjustment 115.799           Mean         30.00029496 min., mg         min., mg         min., mg         min., mg         min., mg         min., mg         1.07           Median         30 Mode         30 Sorption @ 7-12 min., %         Sorption @ 20 min., %         Sorption @ 20 min., %         Max. TGA Wt.         At 20 min., adjustment 115.116           Median         30         Sorption @ 7-12 min., mg         Sorption @ 7-12 min., mg         Sorption @ 20 min., mg         1.07           Median         30         Sorption @ 7-12 min., %         Sorption @ 7-12 min., %         Sorption @ 7-12 min., %         Sorption @ 20 min., %         Min., %         Min., %         O.93         Rel. Error (%) of Parameters of the parameters o	•	-		0.1236
X Intercept as Wt., mg. 116.918   X Intercept as Time, hrs. 20.6   123.7   20.9	3.33 .23	0.00702		nen0120d
as Wt., mg. 116.918 20.6 as Time, hrs. 20.6 123.7 as Time, days 0.9  TEMPERATURE STATISTICS  Max. TGA Wt., (orig data file) 115.799 115.116  Column1  Sorption @ 7-12 sorption @ 20 min., mg min., mg 1.75 1.07  Median 30 Mode 30 Sorption @ 7-12 Sorption @ 20 Standard Deviation 0.040540732 min., % min., % Sample Variance 0.001643551 1.53 0.93  Kurtosis 367.9197048 Skewness -11.59879076 Rel. Error (%) of Range 1.577 7-12 min. Sorp. 4.70.0  Maximum 28.823 -50.7 -70.0	Y Intercept	X Intercept	X Intercept	
TEMPERATURE STATISTICS			•	
TEMPERATURE STATISTICS         Max. TGA Wt., (orig data file) at 115.799         TGA Wt. At 20 min. Adjustment 115.116           Column1         Sorption @ 7-12 min., mg         Sorption @ 20 min., mg min., mg           Mean Standard Error (0.000777476         30 min., mg         1.75 min., mg         1.07           Median 30 Mode 30 Standard Deviation (0.040540732)         Sorption @ 7-12 min., %	116.918		· · · · · · · · · · · · · · · · · · ·	_
TEMPERATURE STATISTICS         (orig data file) 115.799         min. Adjustment 115.116           Column1         Sorption @ 7-12         Sorption @ 20           Mean         30.00029496         min., mg         1.07           Median         30         Sorption @ 7-12         Sorption @ 20         Sorption @ 7-12         Sorption @ 20         min., %         min., %         min., %         min., %         0.93         min., %         0.93         No.93				0.0
Column1         Sorption @ 7-12 Sorption @ 20           Mean         30.00029496 Standard Error         Mode         30.000777476 1.75 1.07         1.75 1.07           Median         30 Mode         30 Sorption @ 7-12 Sorption @ 20         Sorption @ 7-12 Sorption @ 20           Standard Deviation         0.040540732 min., % min., % min., % sample Variance         0.001643551 1.53 0.93         0.93           Kurtosis         367.9197048 Skewness         -11.59879076 Rel. Error (%) of Range         Rel. Error (%) of 7-12 min. Sorp.         Rel. Error (%) of 20 min. Sorp           Minimum         28.823 -50.7 -70.0         -70.0			Max. TGA Wt.,	TGA Wt. At 20
Column1         115.799         115.116           Column1         Sorption @ 7-12         Sorption @ 20           Mean         30.00029496         min., mg         min., mg         1.07           Median         30         Sorption @ 7-12         Sorption @ 20           Median         0.040540732         min., %         Sorption @ 20           Standard Deviation         0.040540732         min., %         min., %         0.93           Kurtosis         367.9197048         Skewness         -11.59879076         Rel. Error (%) of Rel. Error (%) of Range         1.577         7-12 min. Sorp.         20 min. Sorp           Minimum         28.823         -50.7         -70.0           Maximum         30.4	TEMPERATURE STATIST	rics		min. Adjustment
Mean         30.00029496         min., mg         1.07           Median         30         Sorption @ 7-12         Sorption @ 20         Sorption @ 20         Sorption @ 20         min., %         min., %         min., %         min., %         0.93         Min., %         0.93         Min., %         0.93         Min., %         Min., % </td <td></td> <td></td> <td>115.799</td> <td></td>			115.799	
Mean         30.00029496         min., mg         min., mg           Standard Error         0.000777476         1.75         1.07           Median         30         Sorption @ 7-12         Sorption @ 20           Mode         30         Sorption @ 7-12         Sorption @ 20           Standard Deviation         0.040540732         min., %         min., %           Sample Variance         0.001643551         1.53         0.93           Kurtosis         367.9197048         Skewness         -11.59879076         Rel. Error (%) of Rel. Error (%) of Range         1.577         7-12 min. Sorp.         20 min. Sorp           Minimum         28.823         -50.7         -70.0           Maximum         30.4         -50.7         -70.0	Column1			
Standard Error         0.000777476         1.75         1.07           Median         30         Sorption @ 7-12         Sorption @ 20           Mode         30         Sorption @ 7-12         Sorption @ 20           Standard Deviation         0.040540732         min., %         min., %           Sample Variance         0.001643551         1.53         0.93           Kurtosis         367.9197048         Skewness         -11.59879076         Rel. Error (%) of Rel. Error (%) of 7-12 min. Sorp.         Rel. Error (%) of 20 min. Sorp           Minimum         28.823         -50.7         -70.0           Maximum         30.4	Mean	00 00000400		
Median         30         Sorption @ 7-12         Sorption @ 20           Standard Deviation         0.040540732         min., %         min., %           Sample Variance         0.001643551         1.53         0.93           Kurtosis         367.9197048         Skewness         -11.59879076         Rel. Error (%) of Range         Rel. Error (%) of 7-12 min. Sorp.         20 min. Sorp           Minimum         28.823         -50.7         -70.0           Maximum         30.4         -50.7         -70.0			•	_
Mode         30         Sorption @ 7-12         Sorption @ 20           Standard Deviation         0.040540732         min., %         min., %           Sample Variance         0.001643551         1.53         0.93           Kurtosis         367.9197048         Skewness         -11.59879076         Rel. Error (%) of Rel. Error (%) of Range         1.577         7-12 min. Sorp.         20 min. Sorp           Minimum         28.823         -50.7         -70.0           Maximum         30.4			1.75	1.07
Standard Deviation         0.040540732         min., %         min., %           Sample Variance         0.001643551         1.53         0.93           Kurtosis         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048         367.9197048 <t< td=""><td></td><td></td><td>Samtian @ 7 40</td><td>Combine &amp; co</td></t<>			Samtian @ 7 40	Combine & co
Sample Variance       0.001643551       1.53       0.93         Kurtosis       367.9197048       367.9197048       Rel. Error (%) of Rel. Error (%) of Range       11.59879076       Rel. Error (%) of Rel. Error (%) of 20 min. Sorp       20 min. Sorp       20 min. Sorp       -70.0       -70.0         Maximum       30.4       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0       -70.0				•
Kurtosis       367.9197048         Skewness       -11.59879076       Rel. Error (%) of Rel. Error (%) of Panner (%) of Range       1.577       7-12 min. Sorp.       20 min. Sorp         Minimum       28.823       -50.7       -70.0         Maximum       30.4			· ·	
Skewness         -11.59879076         Rel. Error (%) of Rel. Error (%) of Tange         Rel. Error (%) of Tange Properties         Rel. Error (%) of Tange Properties </td <td></td> <td></td> <td>1.55</td> <td>0.93</td>			1.55	0.93
Range       1.577       7-12 min. Sorp.       20 min. Sorp         Minimum       28.823       -50.7       -70.0         Maximum       30.4			Bel Error (%) of	Rel Error (%) of
Minimum 28.823 -50.7 -70.0 Maximum 30.4	Range		(	
Maximum 30.4	•		•	•
^	Maximum		55.,	-70.0
Suiii 81570.802	Sum	81570.802		
Count 2719	Count			
	Confidence Level(95.0%)	0.001524505		
Outilide Level(30,076)	====:(==:70)	0.00.02.7000		

# **DIAMETER STATISTICS**

Column1	
Mean	9.8425
Standard Error	0.01600781
Median	9.845
Mode	9.87
Standard Deviation	0.03201562
Sample Variance	0.001025
Kurtosis	-5.518144
Skewness	-0.0838006
Range	0.06
Minimum	9.81
Maximum	9.87
Sum	39.37
Count	4
Confidence Level(95.0%)	0.05094405

ND/value ND/value A/D

## **THICKNESS STATISTICS**

Column1	
Mean	48.675
Standard Error	0.17969882
Median	48.55
Mode	#N/A
Standard Deviation	0.35939764
Sample Variance	0.12916667
Kurtosis	3.01436004
Skewness	1.6963868
Range	8.0
Minimum	48.4
Maximum	49.2
Sum	194.7
Count	4
Confidence Level(95.0%)	0.57188239

Nordel 2744 pep0130d

# HIGH CONCENTRATION DIFFUSION COEFFICIENT

pep0130d

## **SUMMARY OUTPUT**

Regression Statistics								
Multiple R	0.999591299							
R Square	0.999182765							
Adjusted R Square	0.999147233							
Standard Error	0.004020398							
Observations	25							

## ANOVA

	df	SS	MS	F	Significance F
Regression		0.4545315	0.4545315	28120.6817	5.11105E-37
Residual	2:	3 0.000371763	1.61636E-05		
Total	24	0.454903263			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.994201567	0.003567351	278.694592	4.3336E-42	0.986821948	1.001581186
X Variable 1	-0.019405033	0.000115718	-167.692223	5.111E-37	-0.019644414	-0.019165652

# LOW CONCENTRATION DIFFUSION COEFFICIENT

# SUMMARY OUTPUT

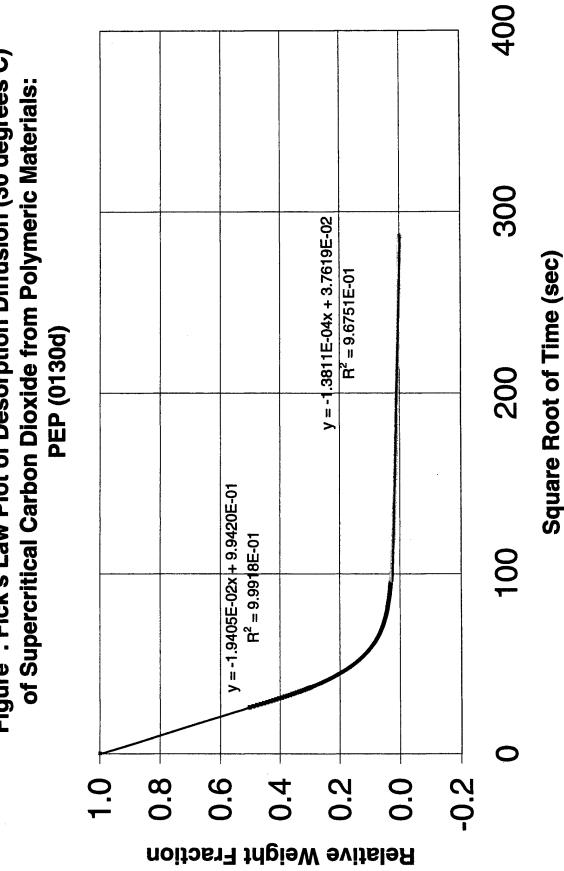
Regression Statistics								
Multiple R	0.983619271							
R Square	0.96750687							
Adjusted R Square	0.967493509							
Standard Error	0.001336773							
Observations	2434							

## ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.129402078	0.129402078	72414.5898	0
Residual	2432	0.00434589	1.78696E-06		
Total	2433	0.133747969			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.03761904	0.000109758	342.7448567	0	0.037403811	0.037834269
X Variable 1	-0.000138113	5.1324E-07	-269.0995908	0	-0.000139119	-0.000137106

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



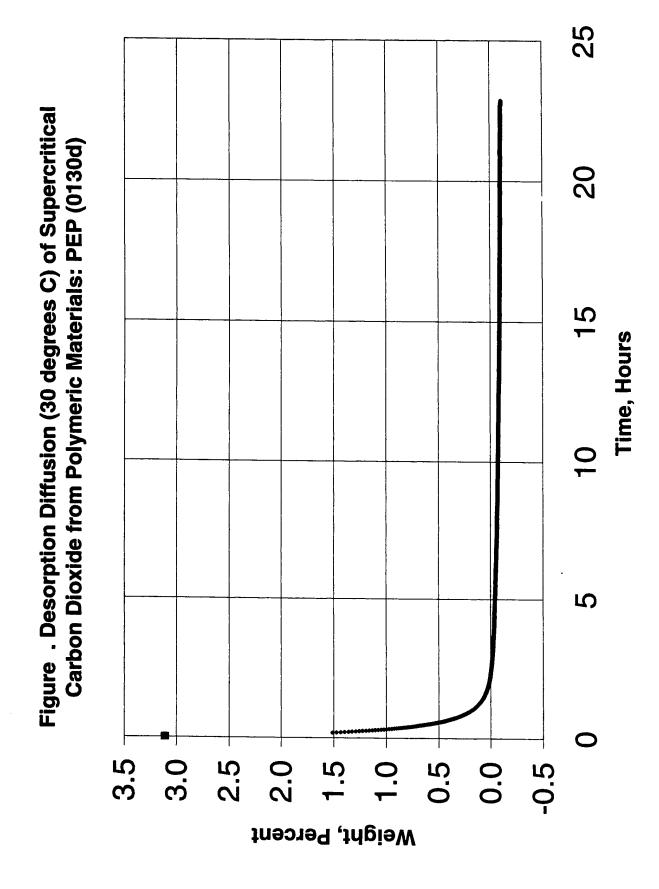


Figure . Desorption Diffusion (30 degrees C) of Supercritical

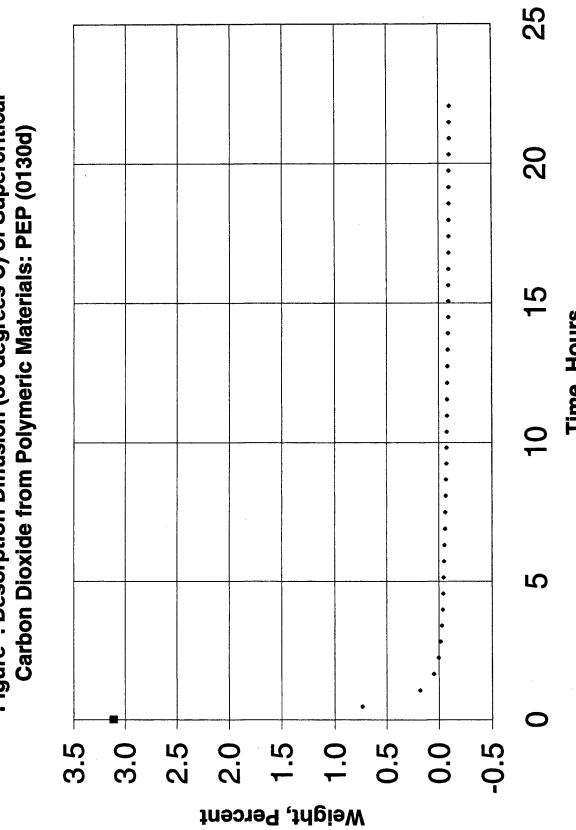
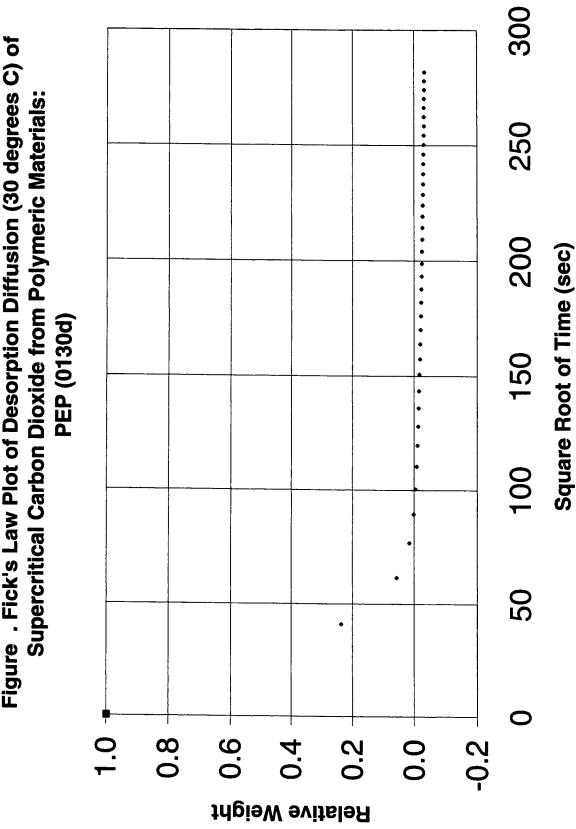


Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of



Hardness Scale N Shore A	Pre Exposure Hardness	93.6	Post Desorption	Hardness	93.9		Change in	Hardness, %	0.32															114,051 ET:10:35										
Adjustment to TGA Time, sec M 635		•	<b>4</b>																					Comment .38"x.04",HP@1400x50,Dry:DS-114.1 M-114.19 TG-114.051 ET:10:35										
TGA Dry Wt., mg L 114.051	of Data	umns	aginning with r								6			ep0130d.01	ž					30D Disk		ermal		0x50,Dry:DS-										
Wt.	for Analysis o	ers refer to col	sters to row, be		_	6	<del>c</del>	() / L4) * 100)	/ (J4 - L4))	)/L4)*100)	N8) / N8) * 10			TA data file : p	14 lines in the parameter block	data	***************************************		luminum Pans	lask SC02 01	Đ.	RPTION-Isoth		к.04",HP@140		÷	ure (øC)	lg)		•				
Wt. @ Zero Time, mg J 117.60	Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n≕4	Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	In = ((Cn - L4) / (J4 - L4))	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8) / N8) * 100)			Source binary TA data file : pep0130d.01	14 lines in the	3 channels of data		Run 37	Module TGA Aluminum Pans	Sample PEP Mask SCO2 0130D Disk	Size 115.799 mg	Method DESORPTION-Isothermal	Operator ws	Comment .38")	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg	Kcell 1.0000	Date 9-May-00	Time 11:47			
Fractional Wt. Loss I 0.237	0.05/ 0.015 0.001	-0.005	0.008	-0.013	-0.014	-0.016	-0.017	-0.019	-0.020	-0.021	-0.022	-0.023	-0.023	-0.024	-0.025	-0.025	-0.026	-0.027	-0.028	-0.028	-0.029	-0.029	-0.030	-0.030	-0.030	-0.031	-0.031	-0.032	-0.032	-0.032	-0.032	-0.033	-0.033	-0.034
Wt. Loss, % H 0.737	0.048 0.0048	-0.015	-0.026 -0.034	-0.039	-0.045	-0.049	-0.053	-0.059	-0.061	-0.064	-0.068	-0.071	-0.073	-0.075	-0.077	-0.079	-0.082	-0.084	-0.086	-0.088	-0.089	-0.091	-0.092	-0.094	-0.095	-0.096	-0.097	-0.099	6.100 0.100	<del>0</del> .100	<del>.</del> 0.10	-0.103	-0.103	-0.104 4
SQRT Adj. Wt. Loss, Time, sec % G H 41.00 0.737	61.50 76.71 89.37	100.44	110.42	128.05	136.01	143.53	150.67	157.49	164.03	170.32	176.39	182.25	187.93	193.44	198.79	204.01	209.10	214.06	218.91	223.66	228.31	232.87	237.34	241.73	246.04	250.27	254.44	258.53	262.57	266.54	270.46	274.32	278.13	281.89
Adjusted Time, hr F 0.467	1.635 2.218	2.803	3.971	4.555	5.139	5.722	6.306	6.890	7.474	8.058	8.642	9.226	9.810	10.394	10.977	11.561	12.145	12.729	13.312	13.896	14.480	15.064	15.648	16.231	16.815	17.399	17.983	18.567	19.151	19.735	20.319	20.903	21,488	22.072
Adjusted Time, sec E 1680.800	3782.660 5884.700 7986.140	10089.080	12192.200	16396.460	18498.980	20599.640	22700.840	24802.700	26905.460	29007.500	31112,300	33213.860	35315.960	37418.060	39518.960	41620.520	43721.720	45823.160	47923.460	50024.540	52126.940	54229.460	56331.260	58432.760	60533.960	62636.360	64737.200	66839.960	68942.420	71045.900	73148.720	75251.540		79460.600
TGA Time, sec D 1045.800	3147.660 5249.700 7351.140	9454.080	11557.200	15761,460	17863.980	19964.640	22065.840	24167.700	26270.460	28372.500	30477.300	32578.860	34680.960	36783.060	38883.960	40985.520	43086.720	45188.160	47288.460	49389.540	51491.940	53594,460	55696.260	57797.760	59898.960	62001.360	64102.200	66204.960	68307.420	70410.900	72513.720	74616.540	76722.660	78825.600
TGA Wt., mg C 114.891	114.254 114.106 114.056	114.034	114.021	114.006	114.000	113.995	113.990	113.984	113.981	113.978	113.974	113.970	113.968	113.965	113.963	113.961	113.958	113.955	113.953	113.951	113.949	113.947	113.946	113.944	113.943	113.941	113.940	113.938	113.937	113.937	113.936	113.934	113.934	113.932
Temp., deg C B 30.007	9 90 00 00 00 00 00 00 00	30.000	30.000	30.00	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.00	30.000	30.001	30.000	30.001	30.000	30.001	30.00	29.999	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000
TGA Time, min A 17.430	52.461 87.495 122.519	157.568	192.620	262.691	297.733	332.744	367.764	402.795	437.841	472.875	507.955	542.981	578.016	613.051	648.066	683.092	718.112	753.136	788.141	823.159	858.199	893.241	928.271	963.296	998.316	1033.356	1068.370	1103.416	1138.457	1173.515	1208.562	1243.609	1278.711	1313.760

**Supercritical Carbon Dioxide** 

and

Polystyrene-butadiene

**PSBR** 

### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

**Test Reference Number:** 

14-1-1

TGA Filename:

psbr143z

Property Test Specification: ASTM D471/D543

Material Code:

Material Name:

Polystyrene-butadiene

Material Class:

**PSBR** SBR

Material Supplier:

**Smithers Scientific DuPont** 

Monomer Source:

Base Polymer (Lot. No.): Unknown

Exposure Liquid:

Supercritical Carbon Dioxide

Replicate Number:

1

## **Pre-Exposure Data**

Measured Diameter (mm):

8.61

8.54

8.64 8.63

0.072

Mean Diameter: Measured Thickness (mils):

<u>40.70</u>

95% Confidence Level (CL): 40.60 40.90

40.90

Mean Thickness:

40.775 Initial Sample Weight (mg):

70.370

0.239

Initial Durometer Hardness:

85.8

Scale: SHORE

Α

95% Confidence Level (CL):

### Sample Exposure Data

**Exposure Date:** 

05/22/00

Time:

14:02:00

Removal Date:

05/22/00

Time:

14:17:00 Weight of Sample After Exposure (mg):

75.00

Total Exposure Time (h:m:s):

00:15:00

Percent Weight Gain:

6.58

Weight Gain (mg): Final Durometer Hardness After Desorption:

4.63

87.1

Percent Hardness Change:

1.5

## **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.002

95% CL:

0.0006

Time Lapse Between Sample Exposure and Sample Desorption (sec): Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

430

Sample Extracted (mg):

0.52

Solubility Corrected for Extractables (mg):

69.850

Sample Extracted (%):

0.74

Solubility Corrected for Extractables (%):

5.15 7.32

## Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm2/sec): Range Including 0,1 Intercept, Relative Weight Fraction:

7.8964E-07 1.00 - 0.46

-1.9363E-02

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.60 - 0.46

Slope: R Square:

0.99994

Y-axis Intercept:

5.1626E+01 9.9963E-01

Low Concentration Diffusion Coefficient (cm2/sec):

9.3694E-11

Slope:

-2.1092E-04

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.05 - 0.003.8201E+02

R Square:

0.99137

Y-axis Intercept:

8.0573E-02

Wt. @ Zero Time, mg K 75.00	Wt. @ Zero Time, % L 6.580	Adjustment to TGA Time, sec M 430	Hardness Shore Scale N A
TGA Dry Wt., mg 70.370	Equil. Wt. Of Desorb. Sample 69.85	Min. Wt. Used For FDC, mg. 69.850	Pre Exposure Hardness 85.8
High Conc. DC, cm2/sec 7.8964E-07	Low Conc. DC, cm2/sec 9.3694E-11	Total Liquid Sorbed, mg. 4.630	Post Desorption Hardness 87.1
R Square 0.99994	R Square 0.99137		Change in Hardness, % 1.5
X Intercept 51.63 Y Intercept	X Intercept 382.0 Y Intercept		Mean Thickness, cm. 0.1036
0.99963  Y Intercept as Wt., mg. 74.972	0.08057  X Intercept as Time, hrs. 40.5	X Intercept as Time, min. 243.2	psbr143z X Intercept as Time, days
TEMPERATURE STATIS		Max. TGA Wt., (orig data file) 72.951	1.7 TGA Wt. At 20 min. Adjustment 71.683
Column1			
Mean Standard Error Median	30.00189014 0.000306054 30.002	Sorption @ 7-12 min., mg 2.58	Sorption @ 20 min., mg 1.31
Mode Standard Deviation Sample Variance Kurtosis	30.002 0.017544129 0.000307796	Sorption @ 7-12 min., % 3.67	Sorption @ 20 min., % 1.87
Skewness Range Minimum	818.301557 -22.99612249 0.845 29.327	Rel. Error (%) of 7-12 min. Sorp. -44.3	Rel. Error (%) of 20 min. Sorp -71.6
Maximum Sum Count Confidence Level(95.0%)	30.172 98586.211 3286 0.000600077		

## **DIAMETER STATISTICS**

Column1						
Mean	8.605					
Standard Error	0.02254625					
Median	8.62					
Mode	#N/A					
Standard Deviation	0.0450925					
Sample Variance	0.00203333					
Kurtosis	2.41709218					
Skewness	-1.5705422					
Range	0.1					
Minimum	8.54					
Maximum	8.64					
Sum	34.42					
Count	4					
Confidence Level(95.0%)	0.07175229					

ND/value ND/value A/D

# THICKNESS STATISTICS

	Column1	
	Mean	40.775
	Standard Error	0.075
	Median	40.8
	Mode	40.9
	Standard Deviation	0.15
	Sample Variance	0.0225
	Kurtosis	-3.9012346
n	Skewness	-0.3703704
Z	Range	0.3
	Minimum	40.6
	Maximum	40.9
	Sum	163.1
	Count	4
	Confidence Level(95.0%)	0.2386837

Unknown psbr143z

# HIGH CONCENTRATION DIFFUSION COEFFICIENT

psbr143z

### **SUMMARY OUTPUT**

Regression Statistics					
Multiple R	0.999968753				
R Square	0.999937507				
Adjusted R Square	0.999932299				
Standard Error	0.00111621				
Observations	14				

### ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.239229675	0.239229675	192009.759	1.34372E-26
Residual	12		1.24592E-06		
Total	13	0.239244627			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.999631372	0.001060248	942.8274908	1.3651E-30	0.997321289	1.001941455
X Variable 1	-0.019362959	4.41886E-05	-438.1891819	1.3437E-26	-0.019459237	-0.01926668

# LOW CONCENTRATION DIFFUSION COEFFICIENT

## **SUMMARY OUTPUT**

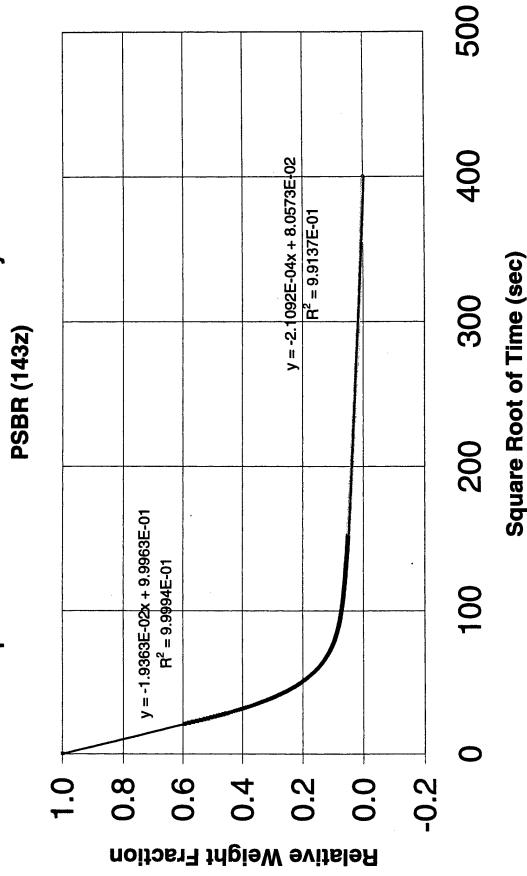
Regression Statistics					
Multiple R	0.995673175				
R Square	0.991365072				
Adjusted R Square	0.991361632				
Standard Error	0.001147306				
Observations	2512				

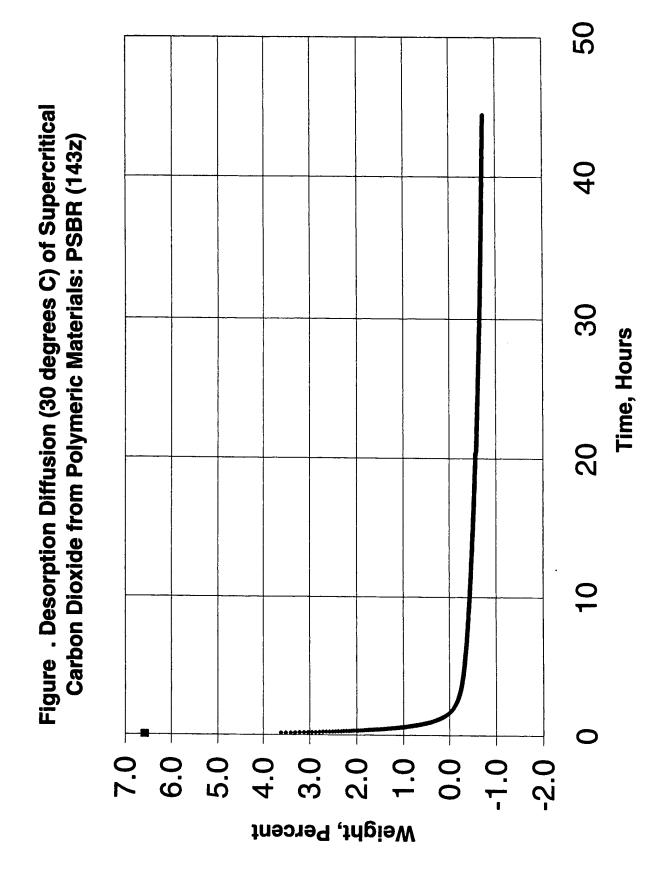
# ANOVA

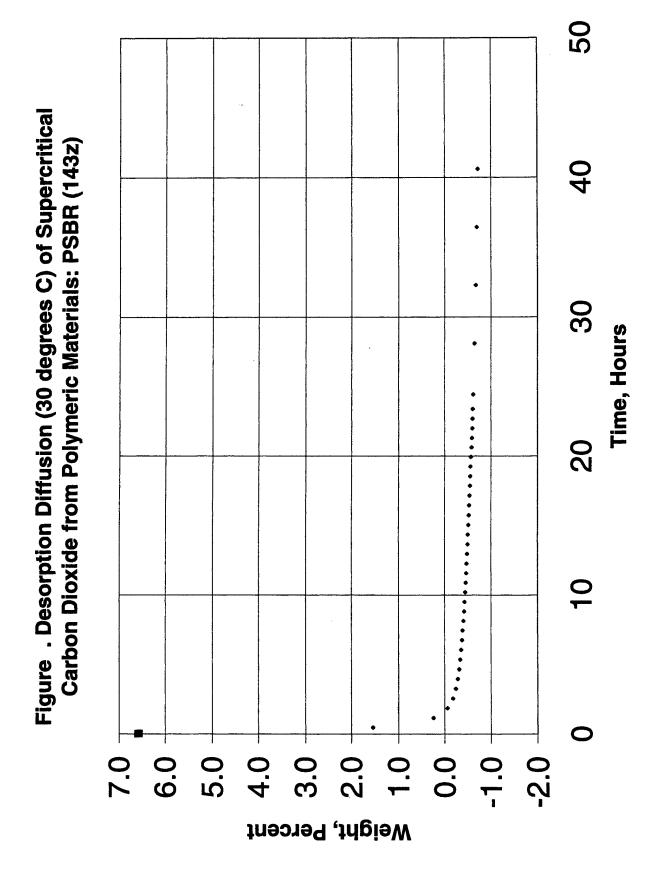
	df	SS	MS	F	Significance F
Regression	1	0.37932119	0.37932119	288169.893	0
Residual	2510	0.003303941	1.31631E-06		_
Total	2511	0.38262513			

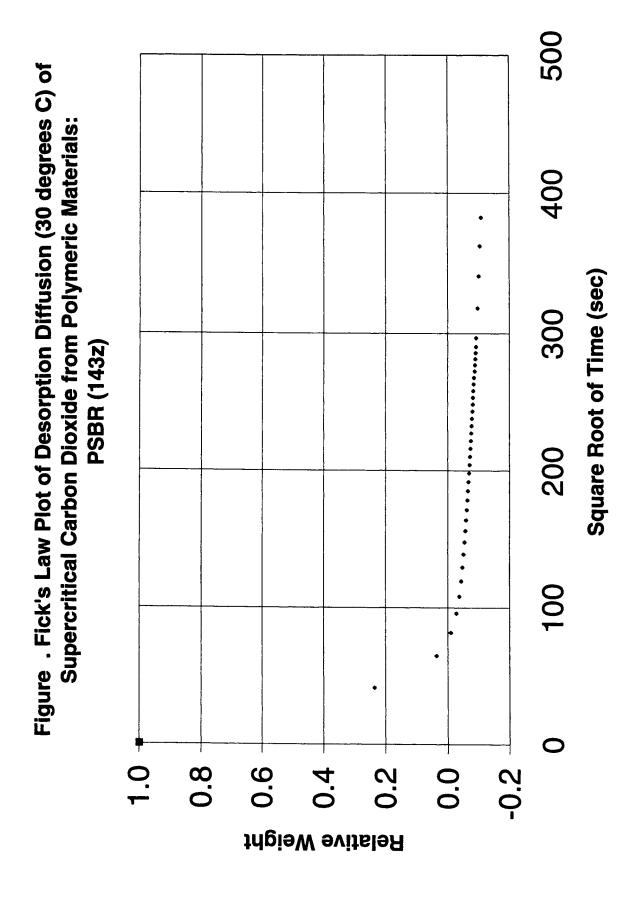
	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.080573277	0.000101198	796.1964679	0	0.080374838	0.080771717
X Variable 1	-0.000210918	3.92907E-07	-536.81458	0	-0.000211689	-0.000210148

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:









Hardness Scale N Shore A Pre Exposure Hardness 85.8	Post Desorption Hardness 87.1 Change in Hardness, %	1.52	M-70.34 TG-70.369 ET: 7:10
Adjustment to TGA Time, sec M 430			
TGA Dry A Wt., mg Tr L 70.369 if Data umns		sbr143z.01 sk sk ne-BUTADIEN	400x50,Dry:DS
Wt. @ Zero Wt. @ Zero TGA Dry Ad Time, mg Time, % Wt., mg TG. L L 75.00 6.581 70.369  Formulae Used for Analysis of Data Uppercase letters refer to columns Lowercase n refers to row, beginning with n=4	Dn = (An * 60) En = (Dn + M4) Fn = (En / 3600) Gn = SQRT (En) Hn = (((Cn - L4) / L4) * 100)	In = ((Cn - L4) / (J4 - L4))  K4 = (((J4 - L4) / L4) * 100)  N16 = (((N12 - N8) / N8) * 100)  Source binary TA data file : psbr143z.01  14 lines in the parameter block  3 channels of data	Comment .339*x.041*,HP@1400x50,Dry:DS-Nsig 3 Sig1 Time (min) Sig2 Temperature (aC) Sig3 Weight (mg) Kcell 1.0000 Date 22-May-00 Time 14:25
	Dn = (An * 60) En = (Dn + M4) Fn = (En / 3600) Gn = SQRT (En) Hn = ((Cn - L4)	In = ((Cn - L4) / (J4 - L4)) K4 = (((J4 - L4) / L4) * 10) N16 = (((N12 - N8) /N8) * Source binary TA data file 14 lines in the parameter 3 channels of data 3 channels of data Module TGA Aluminum P Sample PSBR POLY(ST) Size 72.951 mg Method DESORPTION-Is Operator BSI	Comment .339"x.041",1 Nsig 3 Sig1 Time (min) Sig2 Temperature (øC) Sig3 Weight (mg) Kcell 1.0000 Date 22-May-00 Time 14:25
Fractional Wt. Loss   1   0.236   0.037   -0.009   -0.037   -0.003   -0.003   -0.003   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004   -0.004	0.051 0.051 0.057 0.059 0.059	0.063 0.065 0.067 0.072 0.073 0.075 0.075 0.076 0.078 0.080 0.080	0.085 0.086 0.088 0.089 0.090 0.092 0.093 0.093 0.098 0.098 0.098 0.098 0.098 0.098
Wt. Loss, %	0.310 0.334 0.354 0.372 0.388	0.416 0.429 0.441 0.452 0.463 0.492 0.501 0.510 0.534 0.534 0.534	0.556 0.584 0.579 0.587 0.594 0.605 0.605 0.614 0.643 0.643 0.671
SQRT Adj. Wt. Loss, Time, sec % G H 40.94 1.552 64.63 0.243 81.72 -0.062 95.81 -0.180 108.10 -0.242 119.12 -0.281	129.20 138.56 147.31 155.57 163.43	178.09 184.98 191.62 204.26 210.29 221.88 227.45 232.89 238.20 248.49 253.47	263.16 267.87 272.50 277.05 281.53 286.94 280.28 290.28 296.72 318.07 340.83 362.17
Adjusted Time, hr F 0.465 1.160 1.855 2.550 3.246	4.637 5.333 6.028 6.723 7.419 8.115	8.810 9.505 10.200 10.895 11.590 12.884 12.980 13.676 14.371 15.066 15.761 15.066 17.152 17.152	19.237 19.932 20.627 21.322 22.017 22.712 23.406 24.456 28.102 39.268 36.435
Adjusted Time, sec E 1675.720 4177.120 6677.980 9179.860 11685.100	16693.420 19197.880 21699.940 24203.260 26707.840 29214.460	31716.340 34217.500 36718.840 39221.260 41722.900 44223.460 46727.620 49232.080 51734.380 54236.020 56737.900 64249.420 64249.420	69254.320 71755.480 74257.240 76759.060 79260.160 81761.740 88041.760 101165.880 116166.040
TGA Time, sec Sec D 1245.720 3747.120 6247.980 8749.860 11255.100	16263.420 18767.880 21269.940 23773.260 26277.840	31286.340 33787.500 36288.840 38791.260 41292.900 43793.460 46297.620 51304.380 51304.380 53806.020 56307.900 58811.460 61315.560 633819.420	68824,320 71325,480 73827,240 76329,060 78830,160 81331,740 83833,320 87611,780 1107735,680 115736,040 130736,160
TGA Wt., mg C C 71.461 70.540 70.326 70.42 70.171 70.171	70.151 70.134 70.107 70.096 70.086	70.076 70.067 70.059 70.043 70.029 70.029 70.029 70.010 70.010 70.010 70.010 70.010 70.010 70.010 70.010 70.010 70.010	69.977 69.961 69.956 69.951 69.947 69.943 69.937 69.897 69.897
Temp., deg C B 29.992 30.000 30.002 30.001 30.001 30.001	30.001 30.002 30.002 30.002	30.002 30.002 30.002 30.003 30.002 30.002 30.002 30.002 30.002 30.002 30.002	30.002 30.002 30.004 30.003 30.003 30.003 30.003 30.003 30.003 30.003 30.003
TGA Time, min A 20.762 62.452 104.133 145.831 187.585	271.057 312.798 354.499 396.221 437.964	521.439 563.125 604.814 646.521 688.215 729.891 771.627 813.368 855.073 896.767 938.465 990.191 1021.926	1147.072 1188.758 1230.454 1272.151 1313.836 1355.529 1397.222 1460.196 1678.928 1928.934 2178.936

**Supercritical Carbon Dioxide** 

and

Polyisoprene

PIP

### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

09-1-1

TGA Filename:

pip0131d

Material Name:

Property Test Specification: ASTM D471/D543

Material Code:

PIP

Material Supplier:

Polvisoprene **Smithers Scientific** 

Material Class:

<u>IR</u>

Monomer Source:

Base Polymer (Lot. No.): Unknown

Dupont

Replicate Number:

1

Exposure Liquid:

Supercritical Carbon Dioxide

9.81

41.00

9.85

**Pre-Exposure Data** 

9.83

Mean Diameter:

Measured Diameter (mm):

**Initial Durometer Hardness:** 

9.820

9.79

95% Confidence Level (CL): 41.00

0.041

Measured Thickness (mils): Mean Thickness:

41.000

41.00

41.00 95% Confidence Level (CL):

0.000

Initial Sample Weight (mg):

83.660 66.9

Scale: SHORE

Α

Sample Exposure Data

**Exposure Date:** 

05/10/00

Time:

11:06:00

Removal Date:

05/10/00

Time:

11:21:00

Total Exposure Time (h:m:s):

00:15:00

Weight of Sample After Exposure (mg):

86.50

Weight Gain (mg):

2.84

Percent Weight Gain:

3.39

Final Durometer Hardness After Desorption:

68.7

Percent Hardness Change:

2.7

#### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.001

95% CL:

8000.0

Time Lapse Between Sample Exposure and Sample Desorption (sec): Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

900 83.050

Sample Extracted (mg):

0.61

Solubility Corrected for Extractables (mg):

3.45

Sample Extracted (%):

0.73

Solubility Corrected for Extractables (%):

4.12

#### **Diffusion Coefficient Data: Fick's Law**

High Concentration Diffusion Coefficient (cm2/sec):

8.1549E-07

Range Including 0,1 Intercept, Relative Weight Fraction: Linear Range of Desorption, Relative Weight Fraction:

1.00 - 0.30 0.43 - 0.30

Slope:

R Square:

-1.9569E-02 0.99817

X-axis Intercept: Y-axis Intercept: 5.1365E+01 1.0052E+00

Low Concentration Diffusion Coefficient (cm2/sec):

Linear Range of Desorption, Relative Weight Fraction:

1.2030E-10

Slope:

-2.3768E-04

X-axis Intercept:

0.05 - 0.00 2.9640E+02

R Square:

0.99247

Y-axis Intercept:

7.0450E-02

Wt. @ Zero Time, mg K 86.50	Wt. @ Zero Time, % L 3.395	Adjustment to TGA Time, sec M 900	Hardness Shore Scale N A
TGA Dry Wt., mg 83.660	Equil. Wt. Of Desorb. Sample 83.05	Min. Wt. Used For FDC, mg. 83.050	Pre Exposure Hardness 66.9
High Conc. DC, cm2/sec 8.1549E-07	Low Conc. DC, cm2/sec 1.2030E-10	Total Liquid Sorbed, mg. 2.840	Post Desorption Hardness 68.7
R Square 0.99817	R Square 0.99247		Change in Hardness, % 2.7
X Intercept 51.37	X Intercept 296.4		Mean Thickness, cm.
Y Intercept 1.00519	Y Intercept 0.07045		0.1041
Y Intercept as Wt., mg. 86.949	X Intercept as Time, hrs. 24.4	X Intercept as Time, min. 146.4	pip0131d X Intercept as Time, days 1.0
TEMPERATURE STATIS		Max. TGA Wt., (orig data file) 84.564	TGA Wt. At 20 min. Adjustment 84.159
Column1			
Mean Standard Error Median	30.00092176 0.000420946 30	Sorption @ 7-12 min., mg 0.90	Sorption @ 20 min., mg 0.50
Mode Standard Deviation Sample Variance Kurtosis	30 0.02272328 0.000516347 275.3486451	Sorption @ 7-12 min., % 1.08	Sorption @ 20 min., % 0.60
Skewness Range Minimum	-5.327203032 0.855 29.449	Rel. Error (%) of 7-12 min. Sorp. -68.2	Rel. Error (%) of 20 min. Sorp -82.4
Maximum Sum Count Confidence Level(95.0%)	30.304 87422.686 2914 0.000825382		
2010.(00.070)	2.22002002		

Column1								
Mean	9.82							
Standard Error	0.01290994							
Median	9.82							
Mode	#N/A							
Standard Deviation	0.02581989							
Sample Variance	0.00066667							
Kurtosis	-1.2							
Skewness	-1.858E-13							
Range	0.06							
Minimum	9.79							
Maximum	9.85							
Sum	39.28							
Count	4							
Confidence Level(95.0%)	0.04108524							

ND/value ND/value A/D

#### THICKNESS STATISTICS

Column1	
Mean	41
Standard Error	0
Median	41
Mode	41
Standard Deviation	0
Sample Variance	. 0
Kurtosis	#DIV/0!
Skewness	#DIV/0!
Range	0
Minimum	41
Maximum	41
Sum	164
Count	4
Confidence Level(95.0%)	0

Unknown pip0131d

pip0131d

### **SUMMARY OUTPUT**

Regression Statistics										
Multiple R	0.999086432									
R Square	0.998173699									
Adjusted R Square	0.998033214									
Standard Error	0.00761116									
Observations	15									

### ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.411603256	0.411603256	7105.21296	3.445E-19
Residual	13	0.000753087	5.79298E-05		
Total	14	0.412356343			

		Standard Error		P-value	Lower 95%	Upper 95%
Intercept	1.005190268	0.007455556	134.8243179	7.7337E-22	0.989083521	1.021297014
X Variable 1	-0.019569374	0.000232161	-84.29242527	3.445E-19	-0.020070926	-0.019067821

# LOW CONCENTRATION DIFFUSION COEFFICIENT

# SUMMARY OUTPUT

Regression Statistics										
Multiple R	0.996228405									
R Square	0.992471035									
Adjusted R Square	0.99246815									
Standard Error	0.001134207									
Observations	2612									

	df	SS	MS	F	Significance F
Regression	1	0.442596405	0.442596405	344051.2	0
Residual	2610	0.003357572	1.28643E-06		_
Total	2611	0.445953977			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.070450436	8.98162E-05	784.3847471	0	0.070274318	0.070626555
X Variable 1	-0.000237683	4.05216E-07	-586.5587778	0	-0.000238478	-0.000236888

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:

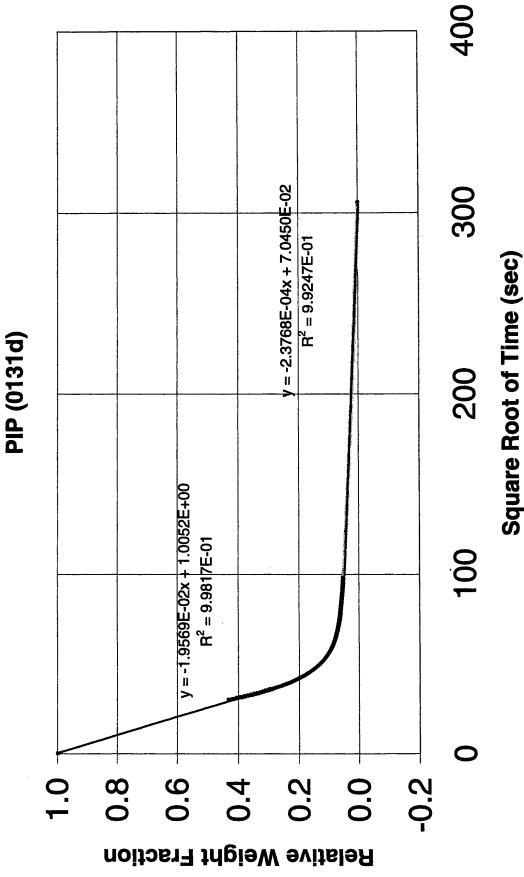
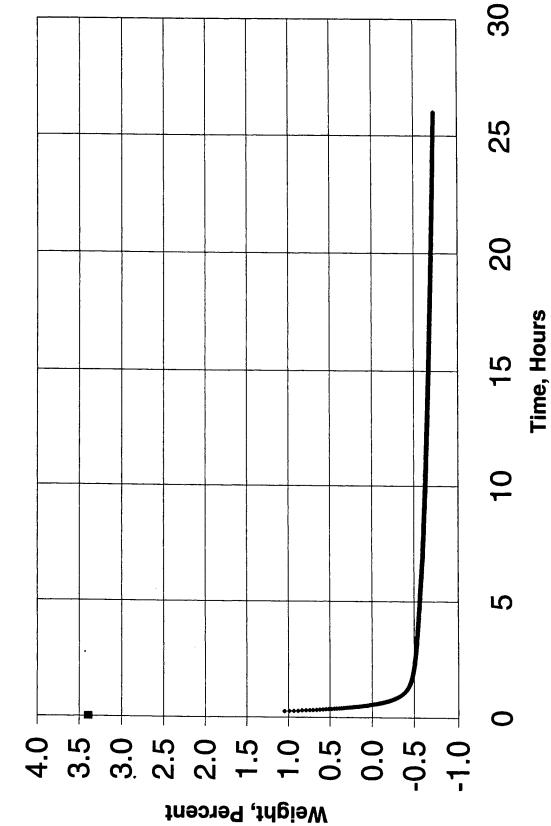


Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PIP (0131d)



25 Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PIP (0131d) 20 15 Time, Hours S Weight, Percent

APPENDIX B

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400 Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: 300 Square Root of Real Time (sec) PIP (0131d) 200 100 9.0 0.8 0.2 0.4 0.0 -0.2 Relative Weight

	nardness	Scale	z	Shore A		Pre Exposure	Hardness	6.99		Post Desorption	Hardness	68.7		Change in	Hardness, %	2.69															3-83.658 E1:											
4 4	Adjustment to	IGA IIMe, sec	Σ	006																											Comment .39"x.043,HP@1400x50,Dry:DS-83.5 M-83.53 1G-83.556 E1:											
	GA DIY	wt., mg	_1	83.658			of Data	olumns	beginning wi	***************************************					_			<b>6</b> 0			pip0131d.0	lock		;		SC	31D Disk	,	hermal	1	400x50,Dry:											
1	Wt. & Zero	"Ime, %	¥	3.397			Formulae Used for Analysis of Data	Uppercase letters refer to columns	.owercase n refers to row, beginning with n=4		ŝ	<b>3</b>	(00	En)	4n = (((Cn - L4) / L4) * 100)	n = ((Cn - L4) / (J4 - L4))	$K4 = (((J4 - L4)/L4)^* 100)$	N16 = (((N12 - N8) / N8) * 100)			Source binary TA data file: pip0131d.01	14 lines in the parameter block	of data			Module TGA Aluminum Pans	Sample PIP Mask SCO2 0131D Disk	mg	Method DESORPTION-Isothermal		9"x.043,HP@1	:	(uiu	ature (øC)	(mg)		Ą					
1	0	lime, mg	ت.	86.50			Formulae Use	Uppercase le	Lowercase n		Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - 1	In = ((Cn - L4	K4 = (((J4 - L	N16 = (((N12			Source binar	14 lines in th	3 channels of data	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	Run 38	Module TGA	Sample PIP I	Size 84.564 mg	Method DES	Operator ws	Comment .39	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 10-May-00	Time 11:36				
	Fractional	Wt. Loss		0.032	-0.118	-0.141	-0.149	-0.154	-0.158	-0.162	-0.165	-0.168	-0.171	-0.174	-0.177	-0.179	-0.180	-0.182	-0.184	-0.186	-0.187	-0.189	-0.190	-0.191	-0.193	-0.194	-0.195	-0.197	-0.198	-0.199	-0.200	-0.201	-0.202	-0.203	-0.204	-0.206	-0.207	-0.207	-0.208	-0.210	-0.210	-0.211
	Wt. Loss,	*	I	0.110	-0.400	-0.478	-0.506	-0.524	-0.538	-0.550	-0.561	-0.571	-0.582	-0.592	-0.600	-0.607	-0.613	-0.619	-0.625	-0.631	-0.636	-0.641	-0.645	-0.650	-0.654	-0.659	-0.663	-0.668	-0.672	-0.676	0.680	-0.684	-0.687	-0.691	-0.694	-0.699	-0.702	-0.705	-0.708	-0.712	-0.715	-0.718
	SOMI Adj. Wt. Loss,	lime, sec	Ø	44.67	64.77	79.98	92.73	103.92	114.04	123.32	131.95	140.05	147.70	154.98	161.93	168.61	175.02	181.20	187.17	192.97	198.60	204.06	209.39	214.58	219.65	224.61	229.46	234.21	238.86	243.43	247.91	252.31	256.65	260.91	265.10	269.22	273.28	277.27	281.22	285.11	288.95	292.73
	Adjusted	lime, hr	L	0.554	1.165	1.777	2.389	3.000	3.613	4.225	4.836	5.448	6.060	6.672	7.284	7.897	8.509	9.120	9.732	10.344	10.956	11.567	12.179	12.790	13.402	14.013	14.625	15.237	15.849	16.461	17.072	17.684	18.296	18.909	19.521	20.133	20.745	21.356	21.968	22.580	23.192	23.803
	Adjusted	Time, sec	ш	1995.000	4195.080	6397.260	8598.900	10800.120	13005.420	15208.860	17410.320	19614.000	21816.300	24019.260	26221.920	28428.720	30632.700	32833.500	35034.420	37237.680	39440.460	41641.500	43842.840	46044.240	48245.640	50448.180	52650.780	54852.780	57055.200	59258.340	61459.380	63661.320	65867.040	68071.860	70275.600	72477.540	74680.320	76880.940	79083.060	81287.580	83490.900	85691.820
i	TGA Time,	<b>20</b> C	۵	1095.000	3295.080	5497.260	7698.900	9900.120	12105.420	14308.860	16510.320	18714.000	20916.300	23119.260	25321.920	27528.720	29732.700	31933.500	34134.420	36337.680	38540.460	40741.500	42942.840	45144.240	47345.640	49548.180	51750.780	53952.780	56155.200	58358.340	60559.380	62761.320	64967.040	67171.860	69375.600	71577.540	73780.320	75980.940	78183.060	80387.580	82590,900	84791.820
i	TGA	Wt., mg	ပ	83.750	83.324	83.258	83.235	83.220	83.208	83.198	83.188	83.180	83.172	83.162	83.156	83.150	83.146	83.140	83.135	83.130	83.126	83.122	83.118	83.114	83.111	83.107	83.103	83.099	83.096	83.093	83.090	83.086	83.083	83.080	83.077	83.074	83.071	83.068	83.066	83.063	83.060	83.057
i	Temp.	o deg C	8	30.015	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.004	30.006	30.001	30.001	30.001	30.001	30.001	30.001	30.001	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30.000
į	TGA Time,	Ē	∢	18.250	54.918	91.621	128.315	165.002	201.757	238.481	275.172	311.900	348.605	385.321	422.032	458.812	495.545	532.225	568.907	605.628	642.341	679.025	715.714	752.404	789.094	825.803	862.513	899.213	935.920	972.639	1009.323	1046.022	1082.784	1119.531	1156.260	1192.959	1229.672	1266.349	1303.051	1339.793	1376.515	1413.197

Blank

#### FIGURE . CHEMICAL-MATERIAL INTERACTION DATA SET:

Supercritical Carbon Dioxide and Polytetrafluoroethylene-propylene TFEP

#### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number: 16-1-1 TGA Filename: tfep153z

Property Test Specification: ASTM D471/D543

Material Name:Polytetrafluoroethylene-propyleneMaterial Code:TFEPMaterial Supplier:Smithers ScientificMaterial Class:FEPM

Monomer Source: Xenox Corp. Base Polymer (Lot. No.): Aflas Exposure Liquid: Supercritical Carbon Dioxide Replicate Number: 1

Pre-Exposure Data

Measured Diameter (mm): 8.48 8.51 8.57 8.47

Mean Diameter: 95% Confidence Level (CL): 0.072

0.092

Measured Thickness (mils): 37.80 37.90 37.80 37.90

Mean Thickness: 37.850 95% Confidence Level (CL):

Initial Sample Weight (mg): 86.660

Initial Durometer Hardness: 91.7 Scale: SHORE A

**Sample Exposure Data** 

Exposure Date: <u>06/01/00</u> Time: <u>10:51:00</u> Removal Date: <u>06/01/00</u> Time: <u>11:06:00</u>

Total Exposure Time (h:m:s): 00:15:00 Weight of Sample After Exposure (mg): 88.60

Weight Gain (mg): 1.94 Percent Weight Gain: 2.24

Final Durometer Hardness After Desorption: 91.2 Percent Weight Gain: 2.24

Percent Weight Gain: 2.24

Percent Hardness Change: -0.5

**Desorption Experiment** 

Mean Temperature of Desorption Experiment (setpoint = 30 deg C): 30.003 95% CL: 0.0007 Time Lapse Between Sample Exposure and Sample Desorption (sec): 435

Equilibrium Weight of Desorbed Sample at 30 deg C (mg): 86.530

Sample Extracted (mg): 0.13 Solubility Corrected for Extractables (mg): 2.07

Sample Extracted (mg): 0.15 Solubility Corrected for Extractables (mg): 2.07
Sample Extracted (%): 0.15 Solubility Corrected for Extractables (%): 2.39

Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm2/sec): 2.2477E-07
Range Including 0,1 Intercept, Relative Weight Fraction: 1.00 - 0.60
Linear Range of Desorption, Relative Weight Fraction: 0.76 - 0.60
Slope: -1.1129E-02 X-axis Intercept: 8.9797E+01

R Square: <u>0.99997</u> Y-axis Intercept: <u>9.9935E-01</u>

Low Concentration Diffusion Coefficient (cm2/sec): 1.5448E-10
Linear Range of Desorption, Relative Weight Fraction: 0.03 - 0.00
Slope: -2.9175E-04 X-axis Intercept: 2.8372E+02

R Square: <u>0.94893</u> Y-axis Intercept: <u>8.2775E-02</u>

Wt. @ Zero Time, mg K 88.60	Wt. @ Zero Time, % L 2.239	Adjustment to TGA Time, sec M 435	Hardness Shore Scale N A
TGA Dry Wt., mg 86.660	Equil. Wt. Of Desorb. Sample 86.53	Min. Wt. Used For FDC, mg. 86.530	Pre Exposure Hardness 91.7
High Conc. DC, cm2/sec 2.2477E-07	Low Conc. DC, cm2/sec 1.5448E-10	Total Liquid Sorbed, mg. 1.940	Post Desorption Hardness 91.2
R Square 0.99997 X Intercept	R Square 0.94893		Change in Hardness, % -0.5
89.80 Y Intercept	X Intercept 283.7 Y Intercept		Mean Thickness, cm. 0.0961
0.99935	0.08277		tfep153z
Y Intercept as Wt., mg. 88.543	X Intercept as Time, hrs. 22.4	X Intercept as Time, min. 134.2	X Intercept as Time, days 0.9
TEMPERATURE STATIST	rics	Max. TGA Wt., (orig data file) 88.120	TGA Wt. At 20 min. Adjustment 87.799
Column1		Sorption @ 7-12	0
Mean Standard Error Median	30.00324974 0.000377758 30.004	min., mg 1.46	Sorption @ 20 min., mg 1.14
Mode Standard Deviation	30.004 0.020381461	Sorption @ 7-12 min., %	Sorption @ 20 min., %
Sample Variance Kurtosis Skewness	0.000415404 744.542963 -21.7623114	1.68 Rel. Error (%) of	1.31 Rel. Error (%) of
Range Minimum Maximum	0.926 29.253 30.179	7-12 min. Sorp. -24.7	20 min. Sorp -41.3
Sum Count	87339.46 2911		
Confidence Level(95.0%)	0.000740701		

Column1								
Mean	8.5075							
Standard Error	0.0225							
Median	8.495							
Mode	#N/A							
Standard Deviation	0.045							
Sample Variance	0.002025							
Kurtosis	0.97607072							
Skewness	1.24828532							
Range	0.1							
Minimum	8.47							
Maximum	8.57							
Sum	34.03							
Count	4							
Confidence Level(95.0%)	0.07160511							

ND/value A/D

ND/value

### THICKNESS STATISTICS

	Column1							
	Mean	37.85						
	Standard Error	0.02886751						
	Median	37.85						
	Mode	37.8						
	Standard Deviation	0.05773503						
	Sample Variance	0.00333333						
	Kurtosis	-6						
Aflas	Skewness	3.6904E-13						
fep153z	Range	0.1						
•	Minimum	37.8						
	Maximum	37.9						
	Sum	151.4						
	Count	4						
	Confidence Level(95.0%)	0.0918694						

A:

tfep153z

### **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.999987226								
R Square	0.999974453								
Adjusted R Square	0.999973572								
Standard Error	0.000396								
Observations	31								

### **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.178008399	0.178008399	1135139.85	3.74453E-68
Residual	29	4.54767E-06	1.56816E-07		
Total	30	0.178012946			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.999351506	0.000304914	3277.48683	2.6367E-82	0.998727887	0.999975125
X Variable 1	-0.01112896	1.04455E-05	-1065.42942	3.7445E-68	-0.011150323	-0.011107596

# LOW CONCENTRATION DIFFUSION COEFFICIENT

# **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.974128452								
R Square	0.948926241								
Adjusted R Square	0.948894538								
Standard Error	0.001909407								
Observations	1613								

	df	SS	MS	F	Significance F
Regression	1	0.109125722	0.109125722	29931.6165	0
Residual	1611	0.005873439	3.64583E-06		
Total	1612	0.114999162			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.082774917	0.000425528	194.5228466	0	0.08194027	0.083609564
X Variable 1	-0.000291753	1.68636E-06	-173.007562	0	-0.00029506	-0.000288445

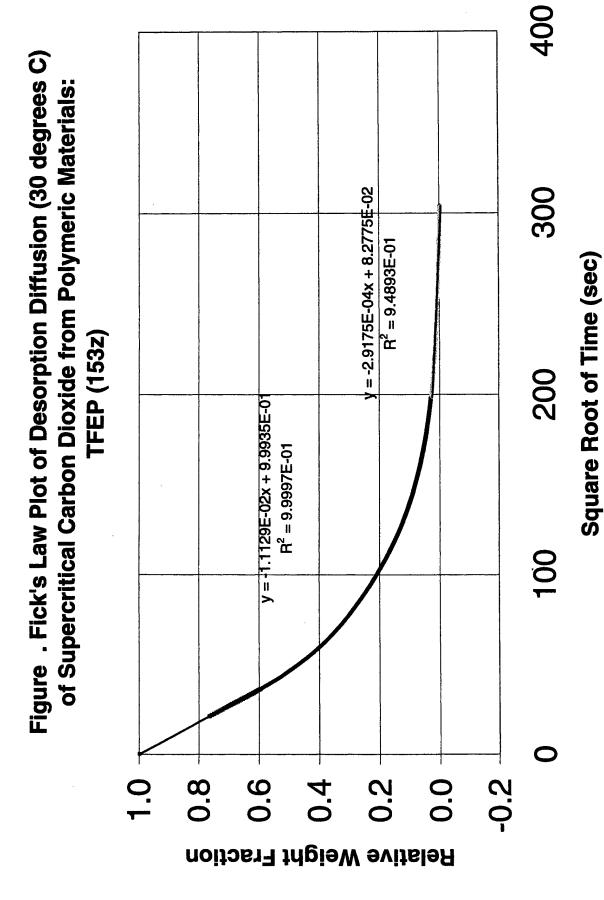


Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: TFEP (153z)

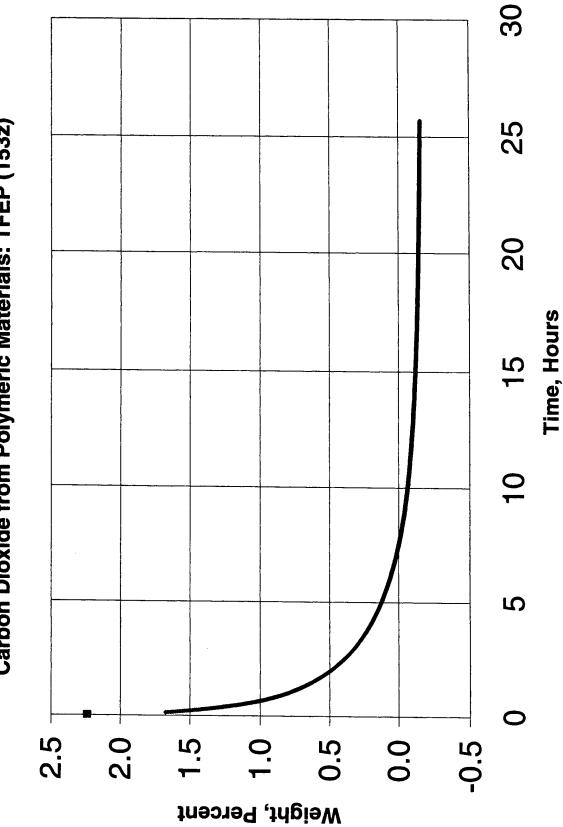
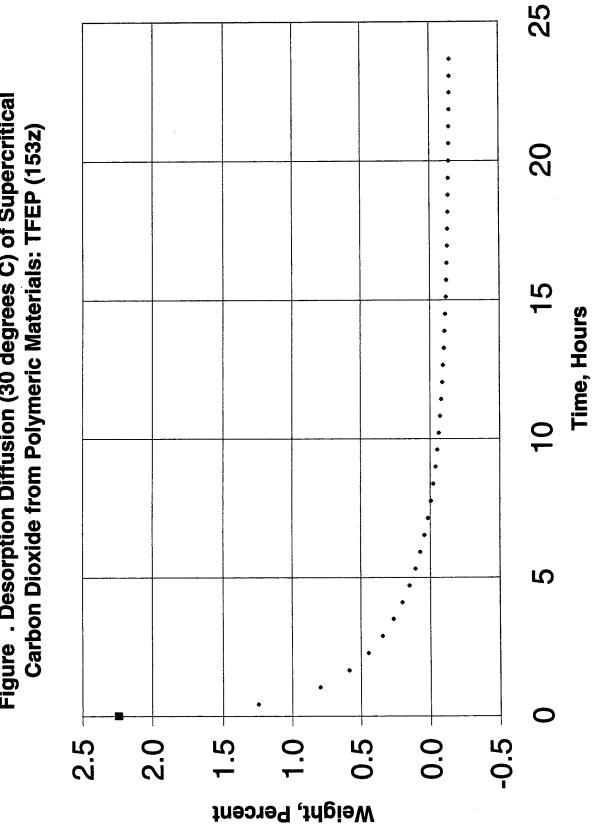
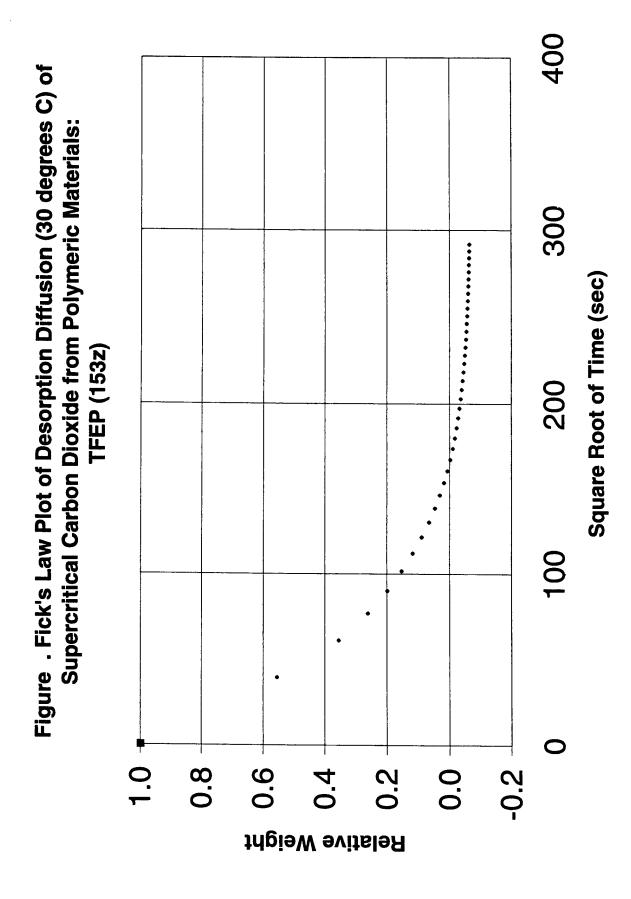


Figure . Desorption Diffusion (30 degrees C) of Supercritical





Hardness	ocaio N	Shore A		Pre Exposure	Hardness	91.7		Post Desorption	Hardness	91.2		Change in	Hardness, %	-0.55															3.657 ET: 7:15											
Adjustment to	iGA lime, sec M	435					vith n=4												_						N				Comment .34"x.040", HP15@1400 Dry: M-86.80 TGA-86.657 ET: 7:15											
TGA Dry	wt., mg -	86.657			s of Data	solumns	beginning v						•		_	<u>@</u>			: tfep153z.0	olock		1		SE SE	Aflas 0153,		thermal		5@1400 Dry											
Wt. @ Zero	% E Y	2.242			d for Analysi	ers refer to c	efers to row,			÷	6	Œ	t)/L4)*100	/ (J4 - L4))	)/L4)*100)	- N8) VN8)			TA data file:	parameter t	data			Juminum Pa	Poly(TFE/P)	5	RPTION-Iso		x.040", HP1		۶	ture (øC)	ng)		_					
Fractional Wt. @ Zero Wt. @ Zero	ger, ag	88.60			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4		Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	$\ln = ((Cn - L4) / (J4 - L4))$	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8) /N8) * 100)			Source binary TA data file: flep153z.01	14 lines in the parameter block	3 channels of data		Run 47	Module TGA Aluminum Pans	Sample TFEP Poly(TFE/P) Affas 0153Z	Size 88.120 mg	Method DESORPTION-Isothermal	Operator bsi	Comment .34"	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 1-Jun-00	Time 11:14				
Fractional	Wi. Loss	0.555	0.355	0.261	0.199	0.153	0.117	0.089	9900	0.047	0.032	0.018	0.007	-0.002	-0.010	-0.017	-0.023	-0.028	-0.032	-0.036	-0.039	-0.042	-0.045	0.048	-0.050	-0.052	-0.053	-0.055	-0.056	-0.057	-0.059	-0.060	-0.061	-0.062	-0.062	-0.063	-0.064	-0.064	-0.065	-0.065
Wt. Loss,	r r	1.243	0.796	0.585	0.445	0.342	0.263	0.200	0.148	0.106	0.071	0.041	0.017	-0.005	-0.023	-0.038	-0.051	-0.063	-0.072	-0.081	-0.088	-0.095	-0.101	-0.107	-0.111	-0.116	-0.119	-0.123	-0.126	-0.129	-0.131	-0.134	-0.136	-0.138	-0.140	-0.141	-0.143	-0.144	-0.145	-0.146
SQRT Adj. Wt. Loss,	Jee sec	39.13	61.09	77.03	90.19	101.67	111.99	121.42	130.18	138.38	146.12	153.46	160.48	167.20	173.66	179.90	185.92	191.75	197.42	202.92	208.27	213.49	218.59	223.57	228.44	233.21	237.88	242.46	246.96	251.38	255.72	259.99	264.19	268.32	272.40	276.41	280.36	284.27	288.12	291.91
Adjusted	Ē L	0.425	1.037	1.648	2.260	2.871	3.484	4.095	4.708	5.319	5.931	6.542	7.154	7.765	8.377	8.990	9.602	10.214	10.826	11.438	12.049	12.661	13.273	13.884	14.496	15.107	15.719	16.330	16.941	17.553	18.165	18.776	19.387	19.999	20.611	21.222	21.834	22.447	23.059	23.670
Adjusted	ime, sec	1531.020	3731.940	5933.580	8134.620	10335.900	12540.720	14743.020	16947.660	19149.480	21350.040	23551.260	25752.720	27954.300	30158.280	32363.100	34565.520	36768.960	38972.820	41175.360	43376.580	45580.080	47781.660	49982.700	52185.300	54385.980	56587.500	58788.240	60989.040	63190.260	65393.040	67593.900	69794.400	71996.100	74199.660	76400.940	78603.300	80807.760		85212.780
TGA Time,	Sec C	1096.020	3296.940	5498.580	7699.620	9900.900	12105.720	14308.020	16512.660	18714.480	20915.040	23116.260	25317.720	27519.300	29723.280	31928.100	34130.520	36333.960	38537.820	40740.360	42941.580	45145.080	47346.660	49547.700	51750.300	53950.980	56152.500	58353.240	60554.040	62755.260	64958.040	67158.900	69359.400	71561.100	73764.660	75965.940	78168.300	80372.760	82575.600	84777.780
TGA	ביי ה	87.735	87.347	87.164	87.043	86.954	86.885	86.830	86.785	86.749	86.718	86.693	86.671	86.653	86.637	86.624	86.613	86.603	86.595	86.587	86.580	86.575	86.569	86.565	86.561	86.557	86.554	86.551	86.548	86.546	86.543	86.541	86.539	86.538	86.536	86.535	86.533	86.532	86.531	86.530
Temp.,		29.995	30.004	30.003	30.005	30.003	30.00	30.001	30.001	30.002	30,001	30.001	30.004	30.004	30.004	30.004	30.004	30.003	30.004	30.003	30.005	30.005	30.005	30.00	30.00	30.00	30.005	30.005	30.005	30.006	30.00	30.004	30.004	30.004	30.004	30.004	30.00	30.003	30.001	30.001
TGA Time,	Ē∢	18.267	54.949	91.643	128.327	165.015	201.762	238.467	275.211	311.908	348.584	385.271	421.962	458.655	495.388	532.135	568.842	605.566	642.297	900.629	715.693	752.418	789.111	825.795	862.505	899.183	935.875	972.554	1009.234	1045.921	1082.634	1119.315	1155.990	1192.685	1229.411	1266.099	1302.805	1339.546	1376.260	1412.963

Blank

#### FIGURE . CHEMICAL-MATERIAL INTERACTION DATA SET:

**Supercritical Carbon Dioxide** 

and

Polyetheretherketone

**PEEK** 

### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

17-1-1

TGA Filename:

peek154z

Property Test Specification: ASTM D471/D543 Polyetheretherketone

Material Code:

PEEK

Material Name: Material Supplier:

Goodfellow Cambridge Ltd.

Material Class:

Blank

Monomer Source:

Unknown

Base Polymer (Lot. No.): Unknown

Replicate Number:

Exposure Liquid:

Supercritical Carbon Dioxide

1

### **Pre-Exposure Data**

Measured Diameter (mm):

8.52

8.39

8.51 8.49

0.095

Mean Diameter: Measured Thickness (mils):

19.40

20.60 20.60

18.10

Mean Thickness:

<u>19.675</u>

31.620

95% Confidence Level (CL):

1.898

Initial Sample Weight (mg): Initial Durometer Hardness:

76.4

Scale: SHORE

D

95% Confidence Level (CL):

#### **Sample Exposure Data**

**Exposure Date:** 

06/02/00

Time:

13:16:00

Removal Date:

06/02/00

Time:

13:31:00

Total Exposure Time (h:m:s):

00:15:00

Weight of Sample After Exposure (mg):

32.00

Weight Gain (mg):

Percent Weight Gain:

1.20

Final Durometer Hardness After Desorption:

0.38

**77.0** 

Percent Hardness Change:

8.0

#### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.001

95% CL:

8000.0

Time Lapse Between Sample Exposure and Sample Desorption (sec): Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

460 31.530

Sample Extracted (mg):

0.09

Solubility Corrected for Extractables (mg):

Sample Extracted (%):

0.28

Solubility Corrected for Extractables (%):

0.47 1.49

#### **Diffusion Coefficient Data: Fick's Law**

High Concentration Diffusion Coefficient (cm2/sec):

7.9890E-08

Range Including 0,1 Intercept, Relative Weight Fraction: Linear Range of Desorption, Relative Weight Fraction: -1.2764E-02

X-axis Intercept:

1.00 - 0.65 0.71 - 0.65

Slope: R Square:

0.99756

Y-axis Intercept:

7.8057E+01 9.9631E-01

Low Concentration Diffusion Coefficient (cm2/sec):

9.4139E-11 0.05 - 0.00

Slope:

-4.3815E-04

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

3.7315E+02

R Square:

0.99517

Y-axis Intercept:

1.6349E-01

Wt. @ Zero Time, mg K 32.00	Wt. @ Zero Time, % L 1.202	Adjustment to TGA Time, sec M 460	Hardness Shore Scale N D
TGA Dry Wt., mg 31.620	Equil. Wt. Of Desorb. Sample 31.53	Min. Wt. Used For FDC, mg. 31.530	Pre Exposure Hardness 76.4
High Conc. DC, cm2/sec 7.9890E-08	Low Conc. DC, cm2/sec 9.4139E-11	Total Liquid Sorbed, mg. 0.380	Post Desorption Hardness 77.0
R Square 0.99756	R Square 0.99517		Change in Hardness, % 0.8
X Intercept 78.06 Y Intercept	X Intercept 373.1 Y Intercept		Mean Thickness, cm. 0.0500
0.99631  Y Intercept as Wt., mg. 31,882	0.16349  X Intercept as Time, hrs. 38.7	X Intercept as Time, min.	peek154z X Intercept as Time, days
TEMPERATURE STATIS		232.1 Max. TGA Wt., (orig data file) 31.868	1.6 TGA Wt. At 20 min. Adjustment 31.809
Column	1	· · · · · · · · · · · · · · · · · · ·	
Mean Standard Error Median	30.00087581 0.000430896 30.001	Sorption @ 7-12 min., mg 0.25	Sorption @ 20 min., mg 0.19
Mode Standard Deviation Sample Variance Kurtosis	29.999 0.024576198 0.000603989 697.3108555	Sorption @ 7-12 min., % 0.78	Sorption @ 20 min., % 0.60
Skewness Range Minimum	-20.17012678 1.14 29.096	Rel. Error (%) of 7-12 min. Sorp. -34.7	Rel. Error (%) of 20 min. Sorp -50.3
Maximum Sum Count Confidence Level(95.0%)	30.236 97592.849 3253 0.000844855		

Column1	Column1								
Mean	8.4775								
Standard Error	0.02982588								
Median	8.5								
Mode	#N/A								
Standard Deviation	0.05965177								
Sample Variance	0.00355833								
Kurtosis	3.09087419								
Skewness	-1.749027								
Range	0.13								
Minimum	8.39								
Maximum	8.52								
Sum	33.91								
Count	4								
Confidence Level(95.0%)	0.09491936								

# THICKNESS STATISTICS

	Column1	
	Mean	19.675
	Standard Error	0.59634302
	Median	20
	Mode	20.6
	Standard Deviation	1.19268604
	Sample Variance	1.4225
	Kurtosis	-0.941917
Unknown	Skewness	-0.9214039
peek154z	Range	2.5
•	Minimum	18.1
	Maximum	20.6
	Sum	78.7
	Count	4
	Confidence Level(95.0%)	1.89783143

ND/value ND/value A/D

peek154z

### **SUMMARY OUTPUT**

Regression Statistics									
Multiple R	0.998781593								
R Square	0.997564671								
Adjusted R Square	0.997321138								
Standard Error	0.004906673								
Observations	12								

### **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.098618322	0.098618322	4096.22143	2.11023E-14
Residual	10	0.000240754	2.40754E-05		
Total	11	0.098859077			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.996311686	0.004754227	209.5633309	1.5049E-19	0.985718606	1.006904766
X Variable 1	-0.01276392	0.000199431	-64.00172992	2.1102E-14	-0.01320828	-0.012319561

# LOW CONCENTRATION DIFFUSION COEFFICIENT

### **SUMMARY OUTPUT**

Regression Statistics								
Multiple R	0.997584511							
R Square	0.995174856							
Adjusted R Square	0.995170221							
Standard Error	0.001161622							
Observations	1043							

	df	SS	MS	F	Significance F
Regression	1	0.28971378	0.28971378	214703.867	0
Residual	1041	0.001404688	1.34936E-06		_
Total	1042	0.291118469			

		Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.16349385	0.000287599	568.4775202	0	0.162929509	0.16405819
X Variable 1	-0.000438149	9.45589E-07	-463.3614866	0	-0.000440005	-0.000436294

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:

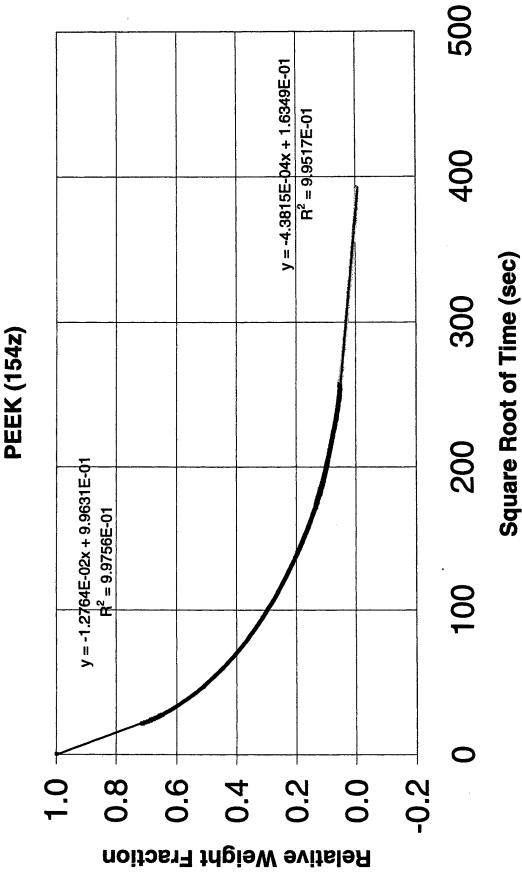
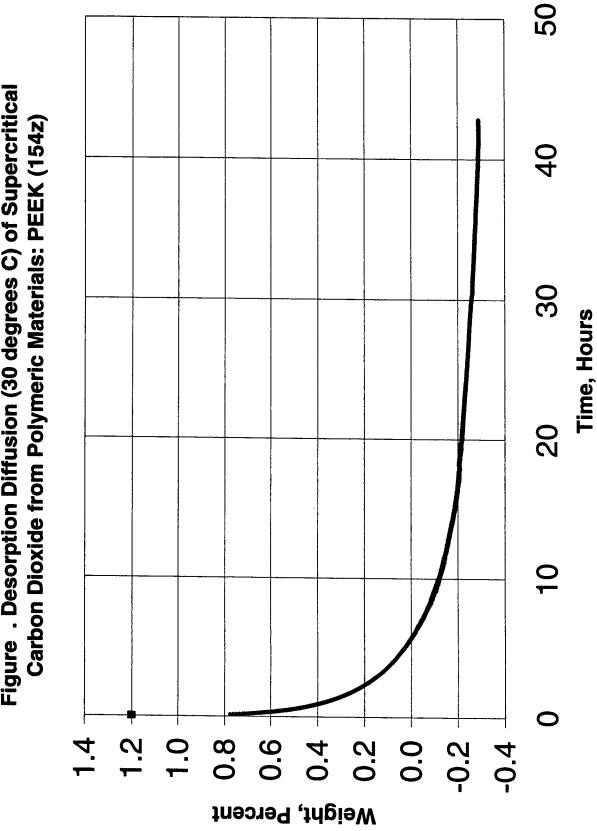
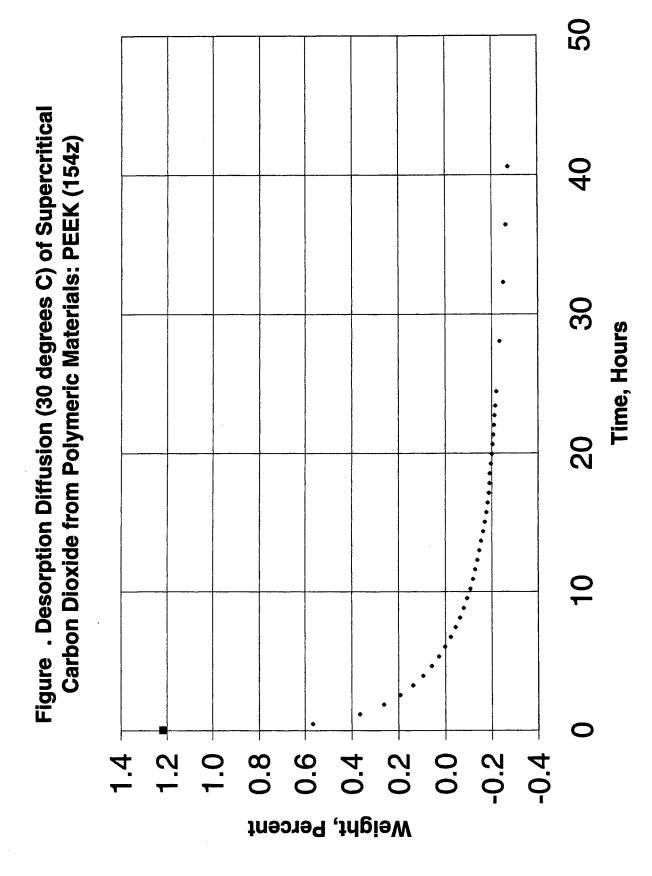
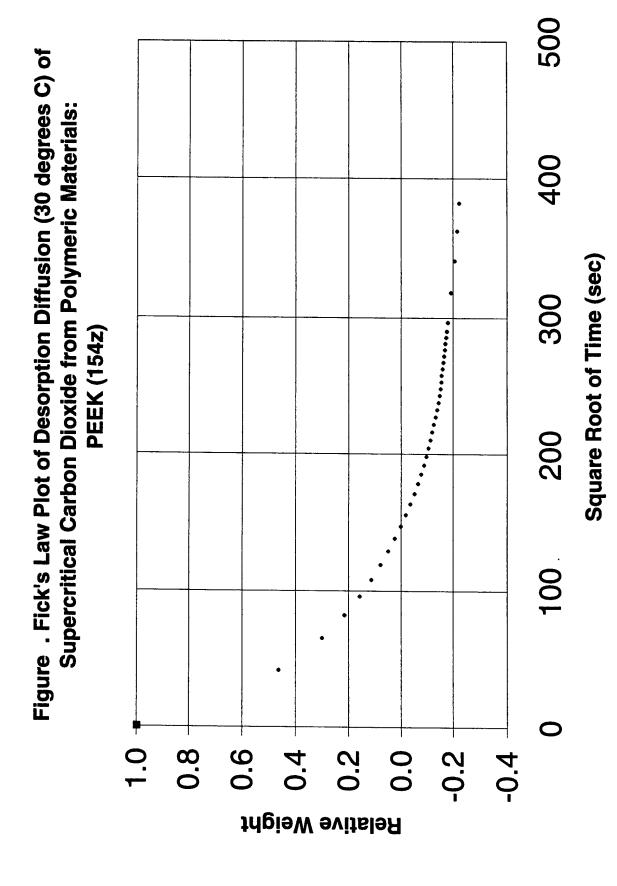


Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PEEK (154z)









Hardness Scale N	Pre Exposure	Hardness 76.4	t o	Post Desorption	Hardness	77.0	į	Change in	nardness, 76	8.0														615 ET: 7:40											
Adjustmentto TGA Time, sec M	9		h n=4	1																				Comment .334"x.020", HP15@1400 Dry: M-31.62 TGA-31.615 ET: 7:40											
TGA Dry Wt., mg L	6.0.10	of Data	oluiriiris beginning wit						_		8	3		peek154z.01	ock 0		•		SI	<b>~</b> 1		hermal	1	5@1400 Dry											
Wt. @ Zero Time, % K	017:1	Formulae Used for Analysis of Data	oppercase retrers refer to continue Lowercase n refers to row, beginning with n≖4	***************	ଚ	14)	(00)	(En)	n = (((Cn - L4) / L4) " 100)	n = ((Cn - L4) / (J4 - L4))	N4 = (((34 - L4) / L4) / (00)	(ONI/ONI = 3		Source binary TA data file: peek154z.01	14 lines in the parameter block	of data			Module TGA Aluminum Pans	Sample PEEK SCO2 0154Z	mg	Method DESORPTION-Isothermal		34"x.020", HP1		rin)	rature (øC)	(mg)		8					
Wt. @ Zero Time, mg J	32.00	Formulae Us	Uppercase in Lowercase n		Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	- (5) Hu = (15)	In = ((Cn - L/	N4 = ((()4 - L	71 NI) = 01 NI		Source binar	14 lines in th	3 channels of data		Run 48	Module TGA	Sample PEE	Size 31.868 mg	Method DES	Operator bsi	Comment .3	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 2-Jun-00	Time 13:39				
Fractional Wt. Loss	0.300 0.216	0.158	0.079	0.049	0.024	0.002	-0.018	-0.036	-0.052	0.065	70.0-	0.096	-0.105	-0.113	-0.120	-0.126	-0.134	-0.140	-0.145	-0.150	-0.154	-0.156	-0.157	-0.161	-0.164	-0.167	-0.169	-0.172	-0.174	-0.177	-0.181	-0.192	-0.206	-0.215	-0.223
SQRT Adj. Wt. Loss, Time, sec % G H	0.365 0.263	0.193	0.096	0.060	0.029	0.00	-0.022	-0.043	0.063	-0.079	\$ 6 6 7 7	9 5	-0.128	-0.138	-0.146	-0.154	-0.163	-0.170	-0.177	-0.182	-0.187	-0.190	-0.191	-0.196	-0.200	-0.203	-0.206	-0.209	-0.212	-0.215	-0.220	-0.234	-0.251	-0.262	-0.272
SQRT Adj. Time, sec G	64.87 81.91	95.99	119.25	129.32	138.66	147.41	155.67	163.51	00.171	178.16	00.00	100 11	204.32	210.35	216.22	221.93	227.50	232.93	238.25	243.44	248.53	253.52	258.40	263.20	267.92	272.55	277.10	281.58	285.99	290.33	296.69	317.96	340.73	362.07	382.23
Adjusted Time, hr F	1.169 1.863	2.559	3.950	4.646	5.340	6.036	6.732	7.427	8.122	8.817	9.512 40.004	10.50	11.597	12.291	12.987	13.682	14.376	15.072	15.767	16.462	17.158	17.853	18.548	19.243	19.939	20.634	21.329	22.024	22.720	23.415	24.452	28.082	32.249	36.416	40.583
Adjusted Time, sec E	4207.900 6708.580	9213.220	14220.940	16723.960	19225.420	21730.360	24233.620	26737.120	29240.680	31742.560	34243.900	30743.000	41748.160	44249.080	46752.160	49254,160	51755.200	54258.100	56761.720	59264.920	61767.940	64271.020	66772.120	69275.140	71779.900	74282.920	76786.000	79287.520	81790.360	84292.420	88026.520	101095.540	116096.380	131096.920	146098.240
TGA Time, sec D	3747.900 6248.580	8753.220	13760.940	16263.960	18765.420	21270.360	23773.620	26277.120	28780.680	31282.560	33/83.900	30707 400	41288.160	43789.080	46292.160	48794.160	51295.200	53798.100	56301.720	58804.920	61307.940	63811.020	66312.120	68815.140	71319.900	73822.920	76326.000	78827.520	81330.360	83832.420	87566.520	100635.540	145636.380	130636.920	145638.240
TGA Wt., mg C	31.730 31.698	31.676	31.645	31.634	31.624	31.616	31.608	31.601	31.595	31.590	200.00	94 570	31.575	31.571	31.569	31.566	31.564	31.561	31.559	31.557	31.556	31.555	31.555	31.553	31.552	31.551	31.550	31.549	31.548	31.547	31.546	31.541	31.536	31.532	31.529
Temp., deg C B	30.000 30.000	30.000	30.00	30.000	30.001	30.001	30.000	30.00	30.001	30.000	50.00	20.00	30.002	30.001	30.002	30.003	30.002	30.001	30.005	30.001	30.002	30.002	30.00	30.002	30.00	30.000	30.001	30.001	30.000	30.002	30.001	30.001	30.002	30.001	30.002
TGA Time, min A	20.766 62.465 104.143	145.887	229.349	271.066	312.757	354.506	396.227	437.952	479.678	521.376	563.065	646.450	688 136	729.818	771.536	813.236	854.920	896.635	938.362	980.082	1021.799	1063.517	1105.202	1146.919	1188.665	1230.382	1272.100	1313.792	1355.506	1397.207	1459.442	1677.259	1927.273	2177.282	2427.304

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#### FIGURE . CHEMICAL-MATERIAL INTERACTION DATA SET:

**Supercritical Carbon Dioxide** 

and

Polydimethylsiloxane

**PDMS** 

#### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

12-1-2

TGA Filename:

pdms139d

Material Name:

Property Test Specification: ASTM D471/D543

Polydimethylsiloxane

Material Supplier:

**Smithers Scientific** 

Material Code: Material Class: **PDMS** MQ

**Monomer Source:** 

Mine Safety, GE

Base Polymer (Lot. No.): Silicone 306017B

**Exposure Liquid:** 

Supercritical Carbon Dioxide

Replicate Number:

Pre-Exposure Data

<u>8.53</u>

8.46 **8.53** 

0.053

Mean Diameter: Measured Thickness (mils):

8.515

46.60

46.90 47.90

Mean Thickness:

46.950

95% Confidence Level (CL):

95% Confidence Level (CL):

1.059

Initial Sample Weight (mg): **Initial Durometer Hardness:** 

Measured Diameter (mm):

77.560 63.9

46.40

8.50

Scale: SHORE

Α

#### **Sample Exposure Data**

**Exposure Date:** 

05/18/00

Time:

13:45:00

Removal Date:

05/18/00

Time:

14:00:00

Total Exposure Time (h:m:s):

00:15:00

Weight of Sample After Exposure (mg):

77.90

Weight Gain (mg):

0.34

Percent Weight Gain:

0.44

Final Durometer Hardness After Desorption:

64.9

Percent Hardness Change:

1.6

#### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

29.999

95% CL:

0.0016

77.580

Time Lapse Between Sample Exposure and Sample Desorption (sec):

<u>495</u>

Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

Solubility Corrected for Extractables (mg):

0.34

Sample Extracted (mg): Sample Extracted (%):

-0.02 -0.03

Solubility Corrected for Extractables (%):

0.44

#### **Diffusion Coefficient Data: Fick's Law**

High Concentration Diffusion Coefficient (cm2/sec):

2.9977E-06

Range Including 0,1 Intercept, Relative Weight Fraction: Linear Range of Desorption, Relative Weight Fraction:

1.00 - 0.20

Slope:

-3.2765E-02

X-axis Intercept:

0.27 - 0.20 3.0531E+01

R Square:

0.99985

Y-axis Intercept:

1.0004E+00

Low Concentration Diffusion Coefficient (cm2/sec):

1.1404E-10

Linear Range of Desorption, Relative Weight Fraction: Slope:

-2.0209E-04

X-axis Intercept:

0.10 - 0.075.4657E+02

R Square:

0.95730

Y-axis Intercept:

1.1046E-01

207

Wt. @ Zero Time, mg K 77.90	Wt. @ Zero Time, % L 0.438	Adjustment to TGA Time, sec M 495	Hardness Shore Scale N A
TGA Dry Wt., mg 77.560	Equil. Wt. Of Desorb. Sample 77.58	Min. Wt. Used For FDC, mg. 77.560	Pre Exposure Hardness 63.9
High Conc. DC, cm2/sec 2.9977E-06	Low Conc. DC, cm2/sec 1.1404E-10	Total Liquid Sorbed, mg. 0.340	Post Desorption Hardness 64.9
R Square 0.99985	R Square 0.95730		Change in Hardness, % 1.6
X Intercept 30.53 Y Intercept	X Intercept 546.6 Y Intercept		Mean Thickness, cm. 0.1193
1.00036	0.11046		
Y Intercept as Wt., mg. 77.928	X Intercept as Time, hrs. 83.0	X Intercept as Time, min. 497.9	pdms139d X Intercept as Time, days 3.5
TEMPERATURE STATIS		Max. TGA Wt., (orig data file) 77.661	TGA Wt. At 20 min. Adjustment 77.605
Column1		0 " 0 = 10	<b>-</b>
Mean Standard Error Median	29.99932403 0.000810539 30	Sorption @ 7-12 min., mg 0.10	Sorption @ 20 min., mg 0.05
Mode Standard Deviation Sample Variance Kurtosis	30.001 0.030305911 0.000918448 473.3664074	Sorption @ 7-12 min., % 0.13	Sorption @ 20 min., % 0.06
Skewness Range Minimum Maximum	-19.01879488 0.988 29.164 30.152	Rel. Error (%) of 7-12 min. Sorp. -70.3	Rel. Error (%) of 20 min. Sorp -86.8
Sum Count Confidence Level(95.0%)	41939.055 1398 0.001590003		

Column1	
Mean	8.505
Standard Error	0.01658312
Median	8.515
Mode	8.53
Standard Deviation	0.03316625
Sample Variance	0.0011
Kurtosis	-0.0495868
Skewness	-1.0964049
Range	0.07
Minimum	8.46
Maximum	8.53
Sum	34.02
Count	4
Confidence Level(95.0%)	0.05277495

### **THICKNESS STATISTICS**

	Column1				
	Mean	46.95			
	Standard Error	0.33291641			
	Median	46.75			
	Mode	#N/A			
	Standard Deviation	0.66583281			
	Sample Variance	0.44333333			
	Kurtosis	2.12030075			
Silicone 306017B	Skewness	1.46348464			
pdms139d	Range	1.5			
	Minimum	46.4			
	Maximum	47.9			
	Sum	187.8			
	Count	4			
	Confidence Level(95.0%)	1.05948958			

ND/value ND/value A/D

pdms139d

#### **SUMMARY OUTPUT**

Regression Statistics								
Multiple R	0.999922767							
R Square	0.99984554							
Adjusted R Square	0.999794054							
Standard Error	0.004940927							
Observations	5							

# ANOVA

	df	SS	MS	F	Significance F
Regression	•	0.474084097	0.474084097	19419.5215	8.14765E-07
Residual	3		2.44128E-05		
Total		0.474157336			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	1.000357561	0.004931748	202.840359	2.6422E-07	0.984662523	1.016052599
X Variable 1	-0.032764957	0.00023512	-139.3539432	8.1477E-07	-0.033513216	-0.032016698

# LOW CONCENTRATION DIFFUSION COEFFICIENT

### **SUMMARY OUTPUT**

Regression Statistics								
Multiple R	0.978415444							
R Square	0.957296781							
Adjusted R Square	0.957262591							
Standard Error	0.001608951							
Observations	1251							

	df	SS	MS	F	Significance F
Regression	1	0.072482686	0.072482686	27999.3805	
Residual	1249		2.58872E-06		
Total	1250	0.075716003			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.110456779	0.000185908	594.1465439	0	0.110092052	0.110821507
X Variable 1	-0.000202092	1.20774E-06	-167.3301541	0	-0.000204461	-0.000199722

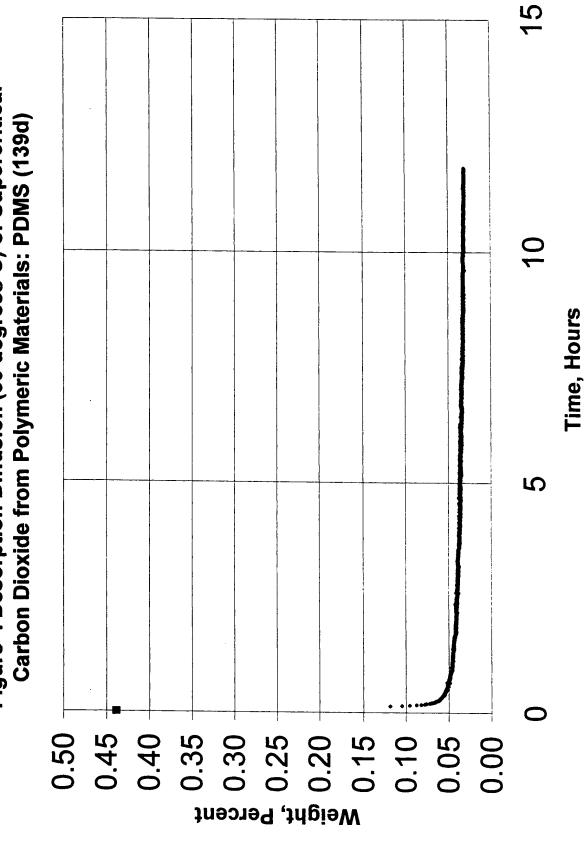
900 Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: -2.0209E-04x + 1.1046E-01 500  $R^2 = 9.5730 \not\models -01$ 400 PDMS (139d) 200 = -3.2765E - 02x + 1.0004E + 00 $R^2 = 9.9985E-01$ 0.6

Square Root of Time (sec)

211

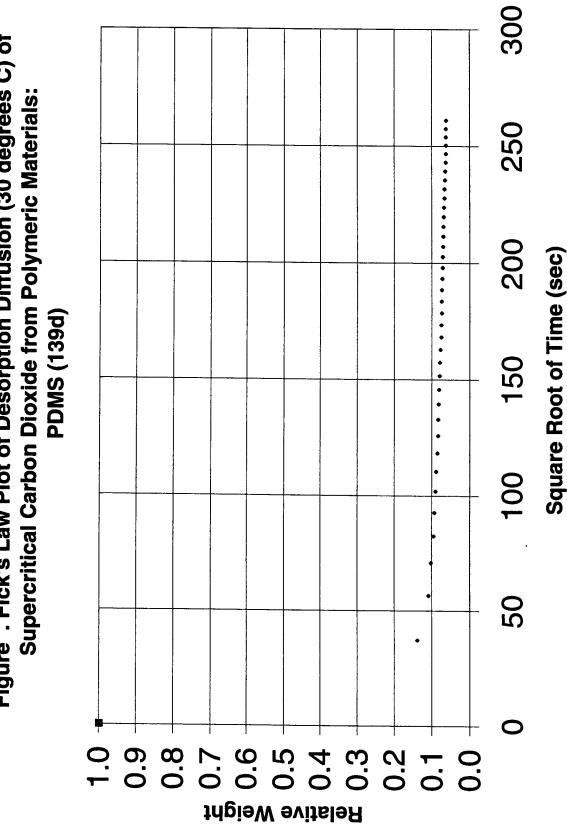
Relative Weight Fraction

Figure . Desorption Diffusion (30 degrees C) of Supercritical



20 Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PDMS (139d) Time, Hours S 9.0 0.5 0.4 0.2 0.0 0.1 Weight, Percent

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



Hardness	Scale	z	Shore A		Pre Exposure	Hardness	63.9		Post Desorption	Hardness	64.9		Change in	Hardness, %	1.56															.77.560 ET: 8:15										
Adjustment to	TGA Time, sec	Z	495					4																						Comment .335"x.047",HP @ 1400x50,Dry:DS-77.5 M-77.50 TG-77.560 ET: 8:15										
TGA Dry	Wt. ma	, 	77.56			f Data	SUUIT	ginning with n	******											dms139d.01	¥					39d Disk		ımal		400x50,Dry:D										
Wt. @ Zero	Time. %	¥	0.438			for Analysis o	ers refer to colo	fers to row, be	*******			•	5	)/L4)*100)	(J4 - L4))	/F4} - 100)	N8) /N8) * 100			「A data file: p	14 lines in the parameter block	data	***************************************		uminum Pans	Glove SC02 1	_	PTION-Isothe		x.047",HP@1		<del>-</del>	ure (øC)	( <u>6</u>		0				
Wt. @ Zero	Time. ma	. J	77.90			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n=4		Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	h = ((Cn - L4) / (J4 - L4))	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8)/N8) * 100)			Source binary TA data file : pdms139d.01	14 lines in the	3 channels of data		Run 42	Module TGA Aluminum Pans	Sample PDMS Glove SC02 139d Disk	Size 77.661 mg	Method DESORPTION-Isothermal	Operator ws	Comment .335	Nsig 3	Sig1 Time (min)	Sig2 Temperat	Sig3 Weight (mg)	Kcell 1.0000	Date 18-May-00	Time 14:09			
Fractional	Wt. Loss	_	0.138	0.109	0.102	0.095	0.093	0.090	0.088	0.085	0.083	0.083	0.082	0.081	0.079	0.079	0.077	0.075	0.075	0.074	0.074	0.073	0.072	0.071	0.071	0.070	0.00	0.069	0.068	0.067	0.066	990.0	0.065	0.064	0.063	0.062	0.062	0.062	0.062	0.062
Wt. Loss.	8	I	0.061	0.048	0.045	0.042	0.041	0.039	0.039	0.037	0.036	0.036	0.036	0.035	0.035	0.034	0.034	0.033	0.033	0.032	0.032	0.032	0.032	0.031	0.031	0.031	0.031	0.030	0.030	0.030	0.029	0.029	0.028	0.028	0.027	0.027	0.027	0.027	0.027	0.027
SORT Adi. Wt. Loss.	Time sec	g	37.28	56.49	70.67	82.44	92.72	101.98	110.46	118.35	125.73	132.71	139.34	145.66	151.72	157.54	163.16	168.59	173.86	178.98	183.95	188.78	193.50	198.09	202.59	206.99	211.30	215.53	219.67	223.73	227.72	231.64	235.50	239.30	243.04	246.71	250.34	253.91	257.44	260.92
Adjusted	Time hr	ш	0.386	0.886	1.387	1.888	2.388	2.889	3.389	3.891	4.391	4.892	5.393	5.894	6.394	6.894	7.394	7.895	8.397	8.838	9.399	9.83	10.400	10.900	11.401	11.901	12.402	12.903	13.404	13.905	14.405	14.905	15.406	15.906	16.408	16.908	17.408	17.909	18.410	18.911
Adjusted	Time sec		1390.140	3191.100	4994.100	6795.540	8596.800	10399.980	12201.900	14007.900	15808.800	17612.100	19415.700	21217.080	23018.160	24818.820	26620.080	28422.360	30228.060	32033.820	33836.520	35637.840	37441.140	39241.560	41043.180	42844.740	44646.240	46451.640	48253.740	50056.440	51857.880	53659.320	55460.460	57263.340	59067.120	60868.140	62669.160	64471.200		68078.640
TGA Time.	Sec	۵	895.140	2696.100	4499.100	6300.540	8101.800	9904.980	11706.900	13512.900	15313.800	17117.100	18920.700	20722.080	22523.160	24323.820	26125.080	27927.360	29733.060	31538.820	33341.520	35142.840	36946.140	38746.560	40548.180	42349.740	44151.240	45956.640	47758.740	49561,440	51362.880	53164.320	54965.460	56768.340	58572.120	60373.140	62174.160	63976.200	65779.800	67583.640
TGA	W	ပ	77.607	77.597	77.595	77.592	77.592	77.591	77.590	77.589	77.588	77.588	77.588	77.587	77.587	77.587	77.586	77.585	77.585	77.585	77.585	77.585	77.585	77.584	77.584	77.584	77.584	77.584	77.583	77.583	77.583	77.582	77.582	77.582	77.581	77.581	77.581	77.581	77.581	77.581
Temp		) ?	29,979	30.000	30.000	30.001	30,000	30.000	30.000	30,000	30.000	30.001	30.000	30.000	30.001	30.000	30.000	30.000	30.001	30.000	30.000	30.001	30.000	30.001	30.000	30.000	30.000	30.000	30,001	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000
TGA Time		4	14.919	44.935	74.985	105.009	135.030	165.083	195.115	225.215	255.230	285.285	315,345	345.368	375.386	405.397	435.418	465.456	495.551	525.647	555.692	585.714	615.769	645.776	675.803	705.829	735.854	765.944	795.979	826.024	856.048	886.072	916.091	946.139	976.202	1006.219	1036.236	1066.270	1096.330	1126.394

Blank

#### FIGURE . CHEMICAL-MATERIAL INTERACTION DATA SET:

Supercritical Carbon Dioxide

and

Polybromobutylene-isoprene

**PBB** 

#### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

10-1-1

TGA Filename:

pbb0138d

Property Test Specification: ASTM D471/D543

Material Code:

Material Name:

Polybromobutylene-isoprene **Smithers Scientific** 

**Material Class:** 

**PBB** BIIR

Material Supplier: Monomer Source:

Miles/Dupont

Base Polymer (Lot. No.): Unknown

Exposure Liquid:

Supercritical Carbon Dioxide

Replicate Number:

# **Pre-Exposure Data**

Measured Diameter (mm): Mean Diameter:

8.50

8.6

<u>8.51</u> 8.52 95% Confidence Level (CL):

0.073

Measured Thickness (mils):

8.515 41.40

41.60

<u>41.60</u>

Mean Thickness: Initial Sample Weight (mg):

41.500

0.184

Initial Durometer Hardness:

68.350 65.9

Scale: SHORE

Α

95% Confidence Level (CL):

41.40

## Sample Exposure Data

**Exposure Date:** 

05/17/00

Time:

10:06:00

Removal Date:

05/17/00

Time: 10:21:00

70.40

Total Exposure Time (h:m:s):

2.05

00:15:00 Weight of Sample After Exposure (mg):

Percent Weight Gain:

3.00

Weight Gain (mg):

Final Durometer Hardness After Desorption:

67.8

Percent Hardness Change:

2.9

#### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C):

30.000

95% CL:

0.0011

Time Lapse Between Sample Exposure and Sample Desorption (sec):

685

Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

68.270

Sample Extracted (mg):

80.0

Solubility Corrected for Extractables (mg):

2.13

Sample Extracted (%):

0.12

Solubility Corrected for Extractables (%):

3.12

### Diffusion Coefficient Data: Fick's Law

High Concentration Diffusion Coefficient (cm2/sec): Range Including 0,1 Intercept, Relative Weight Fraction:

8.4153E-07 <u>1.00 - 0.35</u>

-1.9640E-02

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.49 - 0.355.0890E+01

Slope: R Square:

0.99983

Y-axis Intercept:

9.9947E-01

Low Concentration Diffusion Coefficient (cm2/sec):

7.9684E-11

Slope:

-1.9111E-04

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.04 - 0.002.9056E+02

R Square:

0.98726

Y-axis Intercept:

5.5529E-02

Wt. @ Zero Time, mg K 70.40	Wt. @ Zero Time, % L 2.999	Adjustment to TGA Time, sec M 685	Hardness Shore Scale N A
TGA Dry Wt., mg 68.350	Equil. Wt. Of Desorb. Sample 68.27	Min. Wt. Used For FDC, mg. 68.270	Pre Exposure Hardness 65.9
High Conc. DC, cm2/sec 8.4153E-07	Low Conc. DC, cm2/sec 7.9684E-11	Total Liquid Sorbed, mg. 2.050	Post Desorption Hardness 67.8
R Square 0.99983 X Intercept	R Square 0.98726		Change in Hardness, % 2.9
50.89	X Intercept 290.6		Mean Thickness, cm.
Y Intercept 0.99947	Y Intercept 0.05553		0.1054
Y Intercept as Wt., mg. 70.363	X Intercept as Time, hrs. 23.5	X Intercept as Time, min. 140.7	pbb0138d X Intercept as Time, days 1.0
TEMPERATURE STATIS		Max. TGA Wt., (orig data file) 69.317	TGA Wt. At 20 min. Adjustment 68.966
Column			
Mean Standard Error Median	30.0001569 0.000576801 30	Sorption @ 7-12 min., mg 0.97	Sorption @ 20 min., mg 0.62
Mode Standard Deviation Sample Variance Kurtosis	30 0.030787355 0.000947861 450.4323926	Sorption @ 7-12 min., % 1.41	Sorption @ 20 min., % 0.90
Skewness Range Minimum	-14.59614998 1.259 29.054	Rel. Error (%) of 7-12 min. Sorp. -52.8	Rel. Error (%) of 20 min. Sorp -70.0
Maximum Sum Count Confidence Level(95.0%)	30.313 85470.447 2849 0.001130991		

# **DIAMETER STATISTICS**

Column1									
Mean	8.5325								
Standard Error	0.02286737								
Median	8.515								
Mode	#N/A								
Standard Deviation	0.04573474								
Sample Variance	0.00209167								
Kurtosis	3.3803273								
Skewness	1.81106705								
Range	0.1								
Minimum	8.5								
Maximum	8.6								
Sum	34.13								
Count	4								
Confidence Level(95.0%)	0.07277425								

ND/value ND/value A/D

# **THICKNESS STATISTICS**

	Column1							
	Mean	41.5						
	Standard Error	0.05773503						
	Median	41.5						
	Mode	41.4						
	Standard Deviation	0.11547005						
	Sample Variance	0.01333333						
	Kurtosis	-6						
Unknown	Skewness	0						
pbb0138d	Range	0.2						
	Minimum	41.4						
	<b>M</b> aximum	41.6						
	Sum	166						
	Count	4						
	Confidence Level(95.0%)	0.1837388						

APPENDIX B

# HIGH CONCENTRATION DIFFUSION COEFFICIENT

pbb0138d

### **SUMMARY OUTPUT**

Regression Statistics												
Multiple R 0.99991495												
R Square	0.999829908											
Adjusted R Square	0.999817759											
Standard Error	0.002070178											
Observations	16											

# ANOVA

	df	SS	MS	F	Significance F
Regression		0.352684355	0.352684355	82294.5048	8.62865E-28
Residual	14	5.99989E-05	4.28564E-06		
Total	15	0.352744354			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.999472153	0.001994127	501.2079449	3.4963E-31	0.995195172	1.003749133
X Variable 1	-0.019639835	6.84624E-05	-286.8701881	8.6287E-28	-0.019786673	-0.019492998

# LOW CONCENTRATION DIFFUSION COEFFICIENT

# **SUMMARY OUTPUT**

Regression Statistics										
Multiple R	0.993609146									
R Square	0.987259134									
Adjusted R Square	0.987253995									
Standard Error	0.001120332									
Observations	2481									

# ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.241102733	0.241102733	192091.765	0
Residual	2479	0.003111501	1.25514E-06		
Total	2480	0.244214234			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.055529087	9.65065E-05	575.3920966	0	0.055339846	0.055718329
X Variable 1	-0.000191113	4.36049E-07	-438.2827455	0	-0.000191968	-0.000190258

APPENDIX B

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Relative Weight Fraction

400 Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: 300 y = -1.9111E - 04x + 5.5529E - 02 $R^2 = 9.8726E-01$ PBB (0138d) 200 y = -1.9640E-02x + 9.9947E-01 $R^2 = 9.9983E-01$ 100 0.0 -0.2 0.8 0.2 0.0 0.4

Square Root of Time (sec)

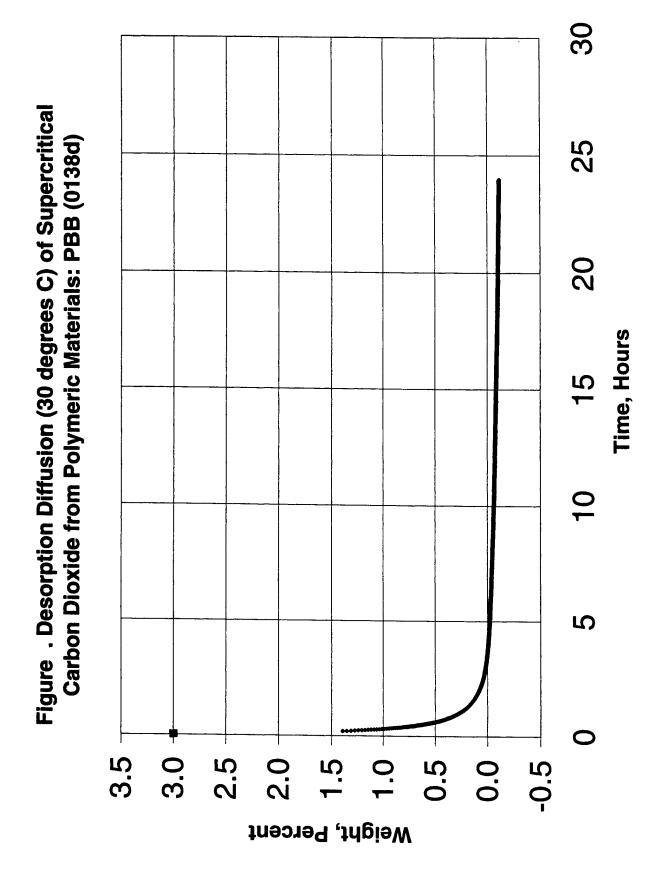


Figure . Desorption Diffusion (30 degrees C) of Supercritical

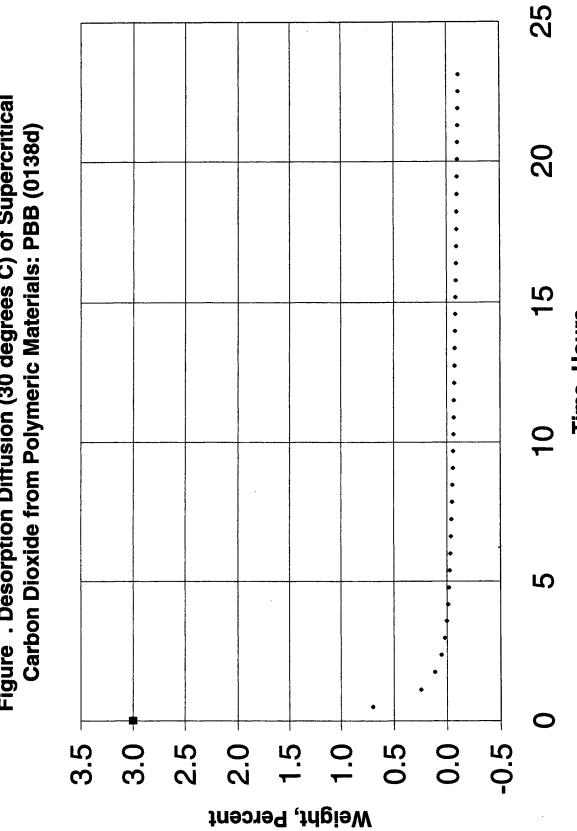
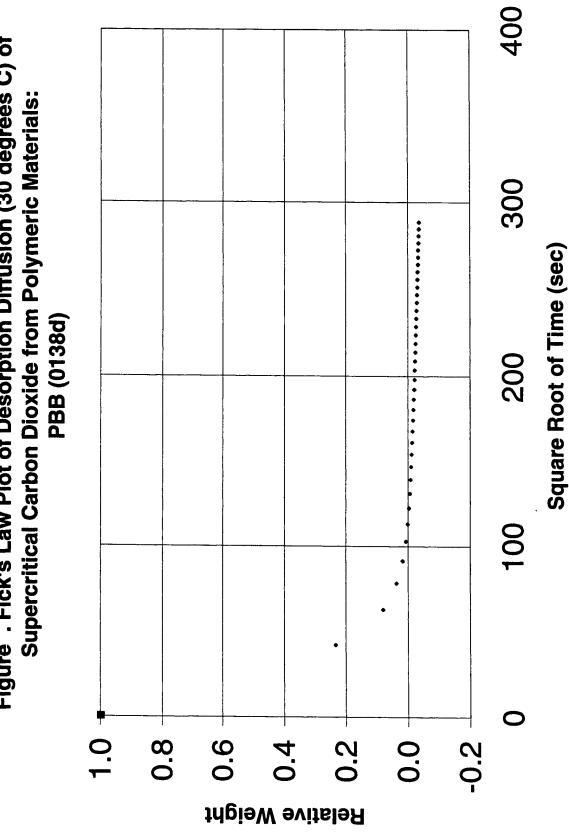


Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



Hardness Scale N Shore A	Pre Exposure Hardness	65.9	Post Desorption	Hardness e7 e	07.0	Change in	Hardness, %	2.88															Comment .336*x.042*,HP@1400x50,Dry:DS-68.4 M-68.28 1G-68.349 E1: 11:25										
Adjustment to TGA Time, sec M 685		7=A																	Disk Disk			1	DS-68.4 M-68.28				à*						
TGA Dry Wt., mg L 68.349	of Data	lumns eginning with							ş	ĝ			ppp0138d.01	ock S				s	CO2 0138D	•	hermal		1400x50,Dry:					_					
Wt. @ Zero Time, % K 3.001	Formulae Used for Analysis of Data	Uppercase letters refer to columns Lowercase n refers to row, beginning with n=4		_ {	æ íg	En.	dn = (((Cn - L4) / L4) * 100)	) / (J4 - L4))	$K4 = (((J4 - L4) / L4)^{-100})$	(001 : (8N/ (8N - ZLN))) = 9LN			Source binary TA data file : pbb0138d.01	14 lines in the parameter block	of data			Module TGA Aluminum Pans	Sample PBrIB-IP 43 Mask SC/22 0138D Disk	gr.	Method DESORPTION-Isothermal		6"x.042",HP®		ii.	ature (øC)	(Bm)		Ş				
Wt. @ Zero Time, mg J 70.40	Formulae Use	Uppercase let		Dn = (An * 60)	En = (Un + M4) Fn = (En / 3600)	Gn = SQRT (En)	Hu = ((Cu - L	In = ((Cn - L4) / (J4 - L4))	K4 = ((()4 - L	N16 = ((N1Z			Source binary	14 lines in th	3 channels of data		Run 41	Module TGA	Sample PBrIE	Size 69.317 mg	Method DES	Operator BSI	Comment .33	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 17-May-00	Time 10:33			
Fractional Wt. Loss 1 0.233	0.081 0.038 0.018	0.007	-0.003	9000	6.008 0.010	-0.012	-0.014	-0.015	-0.017	-0.018	-0.019	-0.020	-0.022	-0.022	-0.023	-0.024	-0.025	-0.026	-0.027	-0.028	-0.028	-0.029	-0.030	-0.031	-0.032	-0.032	-0.033	-0.034	-0.034	-0.035	-0.036	-0.036	-0.037
Wt. Loss, % H 0.699	0.244 0.054 4 4 4	0.022	-0.00	-0.018	6.02 6.03 1.03	-0.036	-0.041	-0.045	-0.050	-0.054	-0.058	.061 -	-0.065	-0.067	-0.070	-0.072	-0.075	-0.078	-0.081	-0.083	-0.085	-0.088	-0.090	-0.093	-0.095	-0.097	-0.099	-0.101	-0.103	-0.105	-0.107	-0.109	- 0.11
SQRT Adj. Wt. Loss, Time, sec % G H 42.19 0.689	63.09 78.63 91.56	102.88	122.43	131.11	139.26	154.26	161.24	167.96	174.39	180.59	186.59	192.40	198.03	203.52	208.86	214.07	219.15	224.13	228.99	233.75	238.41	242.98	247.47	251.87	256.21	260.47	264.67	268.80	272.87	276.87	280.82	284.72	288.56
Adjusted Time, hr F 0.494	1.106 1.717 2.329	2.940	4.163	4.775	5.387 5.999	6.610	7.222	7.836	8.448	9.060	9.671	10.282	10.894	11.505	12.117	12.729	13.341	13.953	14.565	15.177	15.788	16.400	17.011	17.622	18.234	18.846	19.458	20.071	20.683	21.294	21.906	22.517	23.129
	3980.380 6182.080 8383.480	10584.700 12786.820	14988.520	17189.320	19391.980 21595.600	23797.660	25999.600	28209.220	30412.960	32614.540	34815.520	37016.020	39217.840	41418.880	43620.760	45824.140	48027.340	50232.460	52435.120	54636.940	56838.100	59038.600	61239.220	63439.900	65641.840	67844.080	70049.020	72255.280	74457.040	76658.740	78860.380	81062.980	83264.320
TGA Time, sec D 1095.000	3295.380 5497.080 7698.480	9899.700	14303.520	16504.320	18706.980	23112.660	25314.600	27524.220	29727.960	31929.540	34130.520	36331.020	38532.840	40733.880	42935.760	45139.140	47342.340	49547.460	51750.120	53951.940	56153.100	58353.600	60554.220	62754.900	64956.840	67159.080	69364.020	71570.280	73772.040	75973.740	78175.380	80377.980	82579.320
TGA Wt., mg C 68.827	68.516 68.427 68.386	68.364	68.343	68.337	68.332 68.328	68.325	68.321	68.318	68.315	68.312	68.310	68.307	68.305	68.303	68.301	68.300	68.298	68.296	68.294	68.292	68.291	68.289	68.287	68.286	68.284	68.283	68.282	68.280	68.279	68.277	68.276	68.274	68.273
Temp., deg C B 29.999	29.999 30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.00	30.000	30.001	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000
TGA Time, min A 18.250	54.923 91.618 128.308	164.995	238.392	275.072	311.783	385.211	421.910	458.737	495.466	532.159	568.842	605.517	642.214	678.898	715.596	752.319	789.039	825.791	862.502	899.199	935.885	972.560	1009.237	1045.915	1082.614	1119.318	1156.067	1192,838	1229.534	1266.229	1302.923	1339.633	1376.322

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#### FIGURE . CHEMICAL-MATERIAL INTERACTION DATA SET:

**Supercritical Carbon Dioxide** PIB and Polvisobutylene-isoprene

### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number: 11-1-1 TGA Filename: pib0137d

Property Test Specification: ASTM D471/D543

4,

Material Name: Polyisobutylene-isoprene Material Code: PIB Material Supplier:

**Smithers Scientific Material Class:** IIR Monomer Source: Polysar Rubber Base Polymer (Lot. No.): Butyl 301

Supercritical Carbon Dioxide Exposure Liquid: Replicate Number:

**Pre-Exposure Data** 

Measured Diameter (mm): 6.64 6.64 6.62 6.63

Mean Diameter: 95% Confidence Level (CL): 0.015

Measured Thickness (mils): 41.00 41.00 40.40 40.30

Mean Thickness: 40.675 95% Confidence Level (CL): 0.601

Initial Sample Weight (mg): 37.540 **Initial Durometer Hardness:** 66.2 Scale: SHORE Α

Sample Exposure Data

**Exposure Date:** 05/16/00 Time: 13:23:00 Removal Date: 05/16/00 Time: 13:38:00

Total Exposure Time (h:m:s): Weight of Sample After Exposure (mg): 00:15:00 <u>40.50</u>

Weight Gain (mg): 2.96 Percent Weight Gain: 7.88

Final Durometer Hardness After Desorption: 67.7 Percent Hardness Change: 2.3

**Desorption Experiment** 

Mean Temperature of Desorption Experiment (setpoint = 30 deg C): 30.000 95% CL: 0.0014

Time Lapse Between Sample Exposure and Sample Desorption (sec): 545

Equilibrium Weight of Desorbed Sample at 30 deg C (mg): 37.590

Sample Extracted (mg): -0.05 Solubility Corrected for Extractables (mg): 2.96

Sample Extracted (%): -0.13 Solubility Corrected for Extractables (%): 7.88

**Diffusion Coefficient Data: Fick's Law** 

High Concentration Diffusion Coefficient (cm2/sec): 7.7950E-07 Range Including 0,1 Intercept, Relative Weight Fraction: 1.00 - 0.40

Linear Range of Desorption, Relative Weight Fraction: 0.55 - 0.40Slope: -1.9286E-02 X-axis Intercept: 5.1799E+01

R Square: 0.99988 Y-axis Intercept: 9.9897E-01

Low Concentration Diffusion Coefficient (cm2/sec): 3.0844E-11

Linear Range of Desorption, Relative Weight Fraction: 0.04 - 0.02Slope: -1.2131E-04 X-axis Intercept: 3.3677E+02 R Square: 0.75955 Y-axis Intercept: 4.0855E-02

Time, mg	Time, %	TGA Time, sec	Shore Scale
K	L	M	N
40.50	7.885	545	Α
TGA Dry	Equil. Wt. Of	Min. Wt. Used	Pre Exposure
Wt., mg	Desorb. Sample	For FDC, mg.	Hardness
37.540	37.59	37.540	66.2
High Conc.	Low Conc.	Total Liquid	Post Desorption
DC, cm2/sec	DC, cm2/sec	Sorbed, mg.	Hardness
7.7950E-07	3.0844E-11	2.960	67.7
R Square	R Square		Change in
0.99988	0.75955		Hardness, % 2.3
X Intercept	X Intercept		2.0
51.80 ·	336.8		Mean Thickness,
			cm.
Y Intercept	Y Intercept		0.1033
0.99897	0.04085		
			pib0137d
Y Intercept	X Intercept	X Intercept	X Intercept
as Wt., mg.	as Time, hrs.	as Time, min.	as Time, days
40.458	31.5	189.0	1.3
		Max. TGA Wt.,	TGA Wt. At 20
TEMPERATURE STATIST	TICS	(orig data file)	min. Adjustment
		39.176 ´	38.562
Column1			
Mean	00.000	Sorption @ 7-12	Sorption @ 20
Standard Error	29.99950594	min., mg	min., mg
Median	0.000739013	1.64	1.02
Mode	30 29.999	Comtion @ 7.10	Combine @ 00
Standard Deviation	0.029551293	Sorption @ 7-12	Sorption @ 20
Sample Variance	0.000873279	min., % 4.36	min., %
Kurtosis	429.8792472	4.30	2.72
Skewness		Rel. Error (%) of	Rel. Error (%) of
Range	0.988	7-12 min. Sorp.	20 min. Sorp
Minimum	29.192	-44.7	-65.5
Maximum	30.18	~ <del>~~</del> ./	-03.3
Sum	47969.21		
Count	1599		
Confidence Level(95.0%)	0.001449536		

Wt. @ Zero

Adjustment to

Hardness

Wt. @ Zero

# **DIAMETER STATISTICS**

Column1						
Mean	6.6325					
Standard Error	0.00478714					
Median	6.635					
Mode	6.64					
Standard Deviation	0.00957427					
Sample Variance	9.1667E-05					
Kurtosis	-1.2892562					
Skewness	-0.854563					
Range	0.02					
Minimum	6.62					
Maximum	6.64					
Sum	26.53					
Count	4					
Confidence Level(95.0%)	0.01523482					

**THICKNESS STATISTICS** 

	Column1						
	Mean	40.675					
	Standard Error	0.18874586					
	Median	40.7					
	Mode	41					
	Standard Deviation	0.37749172					
	Sample Variance	0.1425					
	Kurtosis	-5.6522007					
Butyl 301	Skewness	-0.0604173					
pib0137d	Range	0.7					
•	Minimum	40.3					
	Maximum	41					
	Sum	162.7					
	Count	4					
	Confidence Level(95.0%)	0.60067413					

ND/value ND/value A/D

# HIGH CONCENTRATION DIFFUSION COEFFICIENT

pib0137d

# **SUMMARY OUTPUT**

Regression Statistics								
Multiple R	0.999940638							
R Square	0.999881279							
Adjusted R Square	0.999872798							
Standard Error	0.00158408							
Observations	16							

### **ANOVA**

	df	SS	MS	F	Significance F
Regression	1	0.295870343	0.295870343	117909.114	6.96392E-29
Residual	14	3.51303E-05	2.50931E-06		
Total	15	0.295905474			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.998966023	0.001504051	664.1835269	6.7905E-33	0.995740151	1.002191895
X Variable 1	-0.019285518	5.61639E-05	-343.3789662	6.9639E-29	-0.019405978	-0.019165058

# LOW CONCENTRATION DIFFUSION COEFFICIENT

# **SUMMARY OUTPUT**

Regression Statistics							
Multiple R	0.871521284						
R Square	0.759549349						
Adjusted R Square	0.759366357						
Standard Error	0.002404989						
Observations	1316						

# **ANOVA**

		SS	MS	<i>r</i>	Significance F
Regression	1	0.024007766	0.024007766	4150.73879	0
Residual	1314	0.007600142	5.78397E-06		
Total	1315	0.031607908			

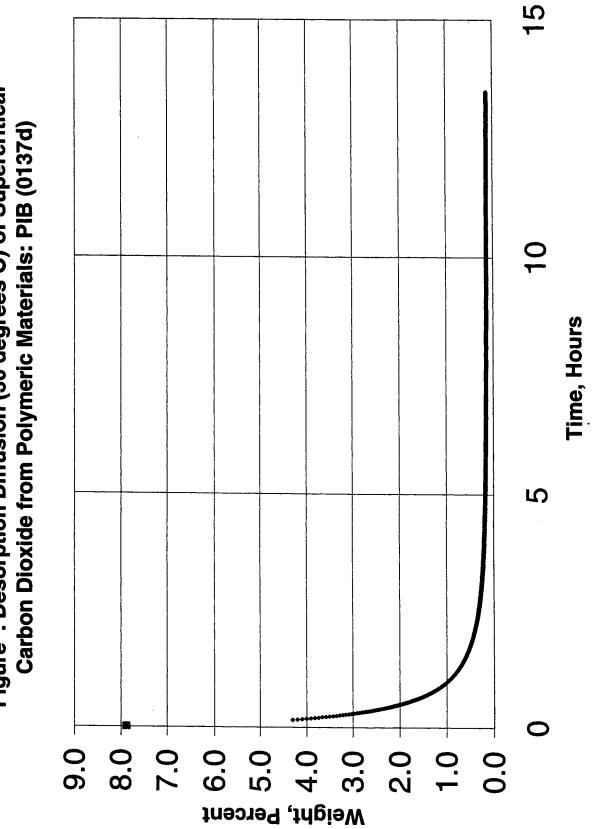
		Standard Error		P-value	Lower 95%	Upper 95%
Intercept	0.040854876	0.000319502	127.8703396	0	0.040228086	0.041481667
X Variable 1	-0.000121314	1.88299E-06	-64.42622748	0	-0.000125008	-0.00011762

400 Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: =-1.2131E-04x + 4.0855E-02  $R^2 = 7.5955E-01$ 300 PIB (0137d) 200 y = -1.9286E-02x + 9.9897E-01 $R^2 = 9.9988E-01$ 100 0.3 0.2 0.1 0.6

Square Root of Time (sec)

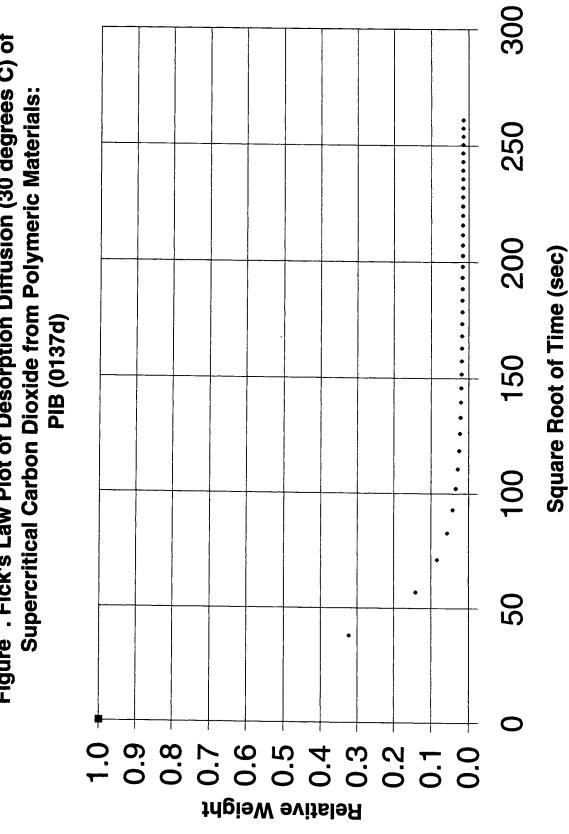
Relative Weight Fraction

Figure . Desorption Diffusion (30 degrees C) of Supercritical



20 Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PIB (0137d) 15 Time, Hours S 1.0 2.0 8.0 6.0 3.0 9.0 Weight, Percent

Figure . Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:



Hardness Scale N Shore A	Pre Exposure	Hardness	66.2	Doet Decomiton	Hardness	67.7		Change in	Hardness, %	2.27															G-37.543 E1: 9:05									
Adjustment to TGA Time, sec M 545			•	η=4																	25"				Comment .263*x.039*, HP @ 1400x50, Dry: DS-37.6 M-37.44 1G-37.543 E1: 9:05									
TGA Dry Wt., mg L 37.543		of Data	lumns	eginning with								Q (			oib0137d.01	S¢.				10	0137D Disk .	•	ermal		1400x50,Dny:									
Wt. @ Zero Time, % K 7.876		Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n rerers to row, beginning with n≕4		æ	6	Œ.	4) / L4) * 100)	/ (J4 - L4))	() / L4) * 100)	N16 = (((N12 - N8) /N8) * 100)			Source binary TA data file : pib0137d.01	14 lines in the parameter block	f data	*******		Module TGA Aluminum Pans	Sample PIB40 Glove SCO2 0137D Disk .25"	Ö	Method DESORPTION-Isothermal		3"x.039",HP@		e E	ture (øC)	ng)		8			
Wt. @ Zero Time, mg J 40.50		Formulae Use	Uppercase lett	Lowercase n r	Dn = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hn = (((Cn - L4) / L4) * 100)	$\ln = ((Cn - L4) / (J4 - L4))$	K4 = (((34 - L4) / L4) * 100)	N16 = (((N12 -			Source binary	14 lines in the	3 channels of data		Run 40	Module TGA A	Sample PIB40	Size 39.176 mg	Method DESC	Operator bsi	Comment .26	Nsig 3	Sig1 Time (min)	Sig2 Temperature (øC)	Sig3 Weight (mg)	Kcell 1.0000	Date 16-May-00	Time 11:49		
Fractional Wt. Loss 1 0.323	0.141	0.057	0.042	0.034 4 000	0.025	0.023	0.021	0.020	0.020	0.019	0.019	0.018	0.018	0.018	0.017	0.017	0.017	0.017	0.017	0.016	0.016	0.016	0.016	0.016	0.016	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015
Wt. Loss, % H 2.543	1.111	0.446	0.332	0.264	0.197	0.181	0.169	0.161	0.155	0.150	0.146	0.143	0.141	0.139	0.137	0.135	0.133	0.132	0.130	0.128	0.127	0.126	0.124	0.123	0.123	0.122	0.121	0.120	0.119	0.118	0.117	0.117	0.116	0.115
SQRT Adj. Wt. Loss, Time, sec % G H 37.95 2.543	56.92	82.72	92.97	102.20	118.52	125.89	132.85	139.46	145.78	151.83	157.66	163.27	168.70	173.97	179.07	184.03	188.86	193.57	198.17	202.66	207.06	211.37	215.58	219.72	223.78	227.78	231.70	235.55	239.35	243.08	246.76	250.38	253.96	257.48 260.95
Adjusted Time, hr F 0.400	0.900	1.901	2.401	2.801	3.902	4.402	4.902	5.403	5.903	6.404	6.904	7.405	7.906	8.407	8.907	9.408	9.908	10.408	10.908	11.409	11.910	12.410	12.910	13.410	13.911	14.412	14.912	15.413	15.913	16.413	16.914	17.414	17.915	18.416 18.916
Adjusted Time, sec E 1439.960	3240.080 5040.980	6842.480	8643.440	10444.520	14047.340	15847.580	17648.060	19449.020	21251.420	23053.340	24856.040	26656.640	28460.360	30264.860	32066.480	33868.400	35669.420	37469.900	39270.500	41073.080	42874.280	44675.360	46476.500	48277.220	50078.540	51882.260	53684.960	55485.560	57286.580	59088.560	60889.520		64495.100	66296.420 68096.780
TGA Time, sec D 894,960	2695.080 4495.980	6297.480	8098.440	44700 060	13502.340	15302.580	17103.060	18904.020	20706.420	22508.340	24311.040	26111.640	27915.360	29719.860	31521.480	33323.400	35124.420	36924.900	38725.500	40528.080	42329.280	44130.360	45931.500	47732.220	49533.540	51337.260	53139.960	54940.560	56741.580	58543.560	60344.520	62146.860	63950.100	65751.420 67551.780
TGA Wt., mg C C 38.498	37.960 37.790	37.711	37.668	37.642	37.617	37.611	37.607	37.603	37.601	37.599	37.598	37.597	37.596	37.595	37.594	37.594	37.593	37.593	37.592	37.591	37.591	37.590	37.590	37.589	37.589	37.589	37.588	37.588	37.588	37.587	37.587	37.587	37.586	37.586 37.586
Temp., deg C B 29.982	30.000	30.000	30.001	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.001	30.000
TGA Time, min A 14.916	44.918 74.933	104.958	134.974	164.992	225.039	255.043	285.051	315.067	345.107	375.139	405.184	435.194	465.256	495.331	525.358	555.390	585.407	615.415	645.425	675.468	705.488	735.506	765.525	795.537	825.559	855.621	885.666	915.676	945.693	975.726	1005.742	1035.781	1065.835	1095.857 1125.863

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#### FIGURE . CHEMICAL-MATERIAL INTERACTION DATA SET:

**Supercritical Carbon Dioxide** 

and

Polystyrene-butadiene

**PSB** 

#### Sorption, Desorption Diffusion, and Indentation Data Sets

#### **Material Data**

Test Reference Number:

13-1-1

TGA Filename:

psb0140d

Property Test Specification: ASTM D471/D543 Material Name:

Material Code:

Material Supplier:

Polystyrene-butadiene The Resinkit Company

**Material Class:** 

**PSB** TP

Monomer Source:

Resin Kit #8

Base Polymer (Lot. No.): Unknown

Exposure Liquid:

Supercritical Carbon Dioxide

Replicate Number:

1

### **Pre-Exposure Data**

Measured Diameter (mm):

9.84

9.74

9.76 9.85 95% Confidence Level (CL):

0.088

Measured Thickness (mils):

Initial Durometer Hardness:

74.70

74.70 76.20

74.50 95% Confidence Level (CL):

1.255

Mean Thickness: Initial Sample Weight (mg):

Mean Diameter:

75.025

142.020

63.6

Scale: SHORE

D

# **Sample Exposure Data**

**Exposure Date:** 

05/19/00

Time:

11:23:00 11:38:00

Removal Date: Total Exposure Time (h:m:s):

05/19/00

Time:

Weight of Sample After Exposure (mg):

148.70

Weight Gain (mg):

00:15:00 6.68

Percent Weight Gain:

4.70

-28.0

Final Durometer Hardness After Desorption:

45.8

Percent Hardness Change:

#### **Desorption Experiment**

Mean Temperature of Desorption Experiment (setpoint = 30 deg C): Time Lapse Between Sample Exposure and Sample Desorption (sec):

30.001

95% CL:

0.0004

Equilibrium Weight of Desorbed Sample at 30 deg C (mg):

<u>610</u> 142.000

Sample Extracted (mg):

0.02

Solubility Corrected for Extractables (mg):

Sample Extracted (%):

0.01

Solubility Corrected for Extractables (%):

6.70 4.72

# **Diffusion Coefficient Data: Fick's Law**

High Concentration Diffusion Coefficient (cm2/sec): Range Including 0,1 Intercept, Relative Weight Fraction: 9.7266E-07 1.00 - 0.54

Slope:

-1.1680E-02

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.67 - 0.54

R Square:

0.99872

Y-axis Intercept:

8.5039E+01 9.9321E-01

Low Concentration Diffusion Coefficient (cm2/sec):

1.4751E-10

Slope:

-1.4383E-04

Linear Range of Desorption, Relative Weight Fraction: X-axis Intercept:

0.02 - 0.00 2.9524E+02

R Square:

0.82883

Y-axis Intercept:

4.2465E-02

Wt. @ Zero Time, mg K 148.70	Wt. @ Zero Time, % L 4.704	Adjustment to TGA Time, sec M 610	Hardness Shore Scale N D
TGA Dry Wt., mg 142.020	Equil. Wt. Of Desorb. Sample 142.00	Min. Wt. Used For FDC, mg. 142.000	Pre Exposure Hardness 63.6
High Conc. DC, cm2/sec 9.7266E-07	Low Conc. DC, cm2/sec 1.4751E-10	Total Liquid Sorbed, mg. 6.680	Post Desorption Hardness 45.8
R Square 0.99872	R Square 0.82883		Change in Hardness, % -28.0
X Intercept 85.04	X Intercept 295.2		Mean Thickness, cm.
Y Intercept 0.99321	Y Intercept 0.04247		0.1906
Y Intercept as Wt., mg. 147.691	X Intercept as Time, hrs. 24.2	X Intercept as Time, min. 145.3	psb0140d X Intercept as Time, days 1.0
TEMPERATURE STATIS		Max. TGA Wt., (orig data file) 146.740	TGA Wt. At 20 min. Adjustment 145.935
Column1			
Mean	00 0000047	Sorption @ 7-12	Sorption @ 20
Standard Error	30.0009317 0.000200313	min., mg	min., mg
Median	30	4.72	3.91
Mode		Sorption @ 7-12	Sorption @ 20
Standard Deviation	0.011080771	min., %	min., %
Sample Variance	0.000122783	3.32	2.76
Kurtosis	266.4820057	5.62	20
Skewness	15.10275641	Rel. Error (%) of	Rel. Error (%) of
Range	0.281	7-12 min. Sorp.	20 min. Sorp
Minimum	29.943	<b>-2</b> 9.3	-41.4
Maximum	30.224		
Sum	91802.851		
Count	3060		
Confidence Level(95.0%)	0.000392762		

# **DIAMETER STATISTICS**

Column1	
Mean	9.7975
Standard Error	0.02780138
Median	9.8
Mode	#N/A
Standard Deviation	0.05560276
Sample Variance	0.00309167
Kurtosis	-5.2112089
Skewness	-0.0828945
Range	0.11
Minimum	9.74
Maximum	9.85
Sum	39.19
Count	4
Confidence Level(95.0%)	0.08847648

# **THICKNESS STATISTICS**

	Column1							
	Mean	75.025						
	Standard Error	0.39449335						
	Median	74.7						
	Mode	74.7						
	Standard Deviation	0.78898669						
	Sample Variance	0.6225						
	Kurtosis	3.74193965						
Unknown	Skewness	1.91237109						
psb0140d	Range	1.7						
•	Minimum	74.5						
	Maximum	76.2						
	Sum	300.1						
	Count	4						
	Confidence Level(95.0%)	1.25545507						

ND/value ND/value A/D

# HIGH CONCENTRATION DIFFUSION COEFFICIENT

psb0140d

### **SUMMARY OUTPUT**

Regression S	tatistics
Multiple R	0.999359786
R Square	0.998719982
Adjusted R Square	0.998672574
Standard Error	0.003081805
Observations	29

# ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.200079147	0.200079147	21066.4595	1.34869E-40
Residual	27	0.000256433	9.49752E-06		
Total	28	0.20033558			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.993213175	0.002687177	369.6121544	1.4976E-51	0.987699547	0.998726802
X Variable 1	-0.011679524	8.04691E-05	-145.1428934	1.3487E-40	-0.011844633	-0.011514415

# LOW CONCENTRATION DIFFUSION COEFFICIENT

# **SUMMARY OUTPUT**

Regression Statistics													
Multiple R	0.910403271												
R Square	0.828834116												
Adjusted R Square	0.828738332												
Standard Error	0.002281732												
Observations	1789												

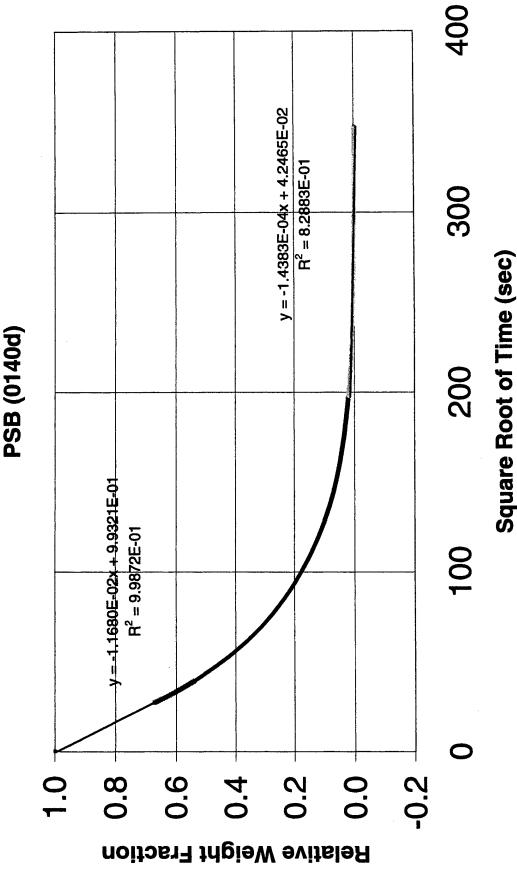
# ANOVA

	df	SS	MS	F	Significance F
Regression	1	0.045050986	0.045050986	8653.16461	
Residual	1787	0.009303661	5.2063E-06		_
Total	1788	0.054354647			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.04246542	0.000400887	105.9286262	0	0.041679163	0.043251677
X Variable 1	-0.000143832	1.54621E-06	-93.02238766	0	-0.000146864	-0.000140799

Figure. Fick's Law Plot of Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials:

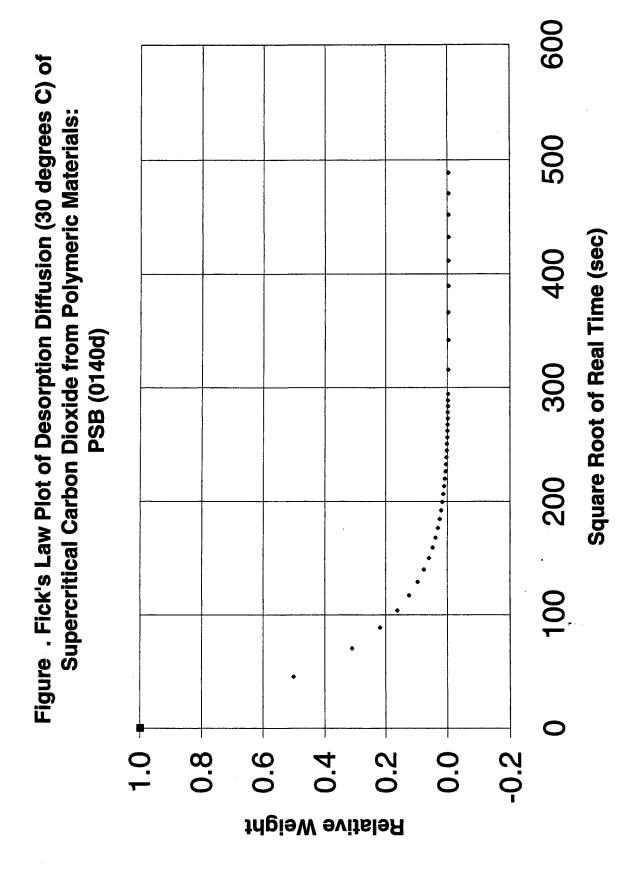
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40 Figure . Desorption Diffusion (30 degrees C) of Supercritical Carbon Dioxide from Polymeric Materials: PSB (0140d) 30 Time, Hours 20 5.0 4.0 3.0 2.0 1.0 0.0 Weight, Percent

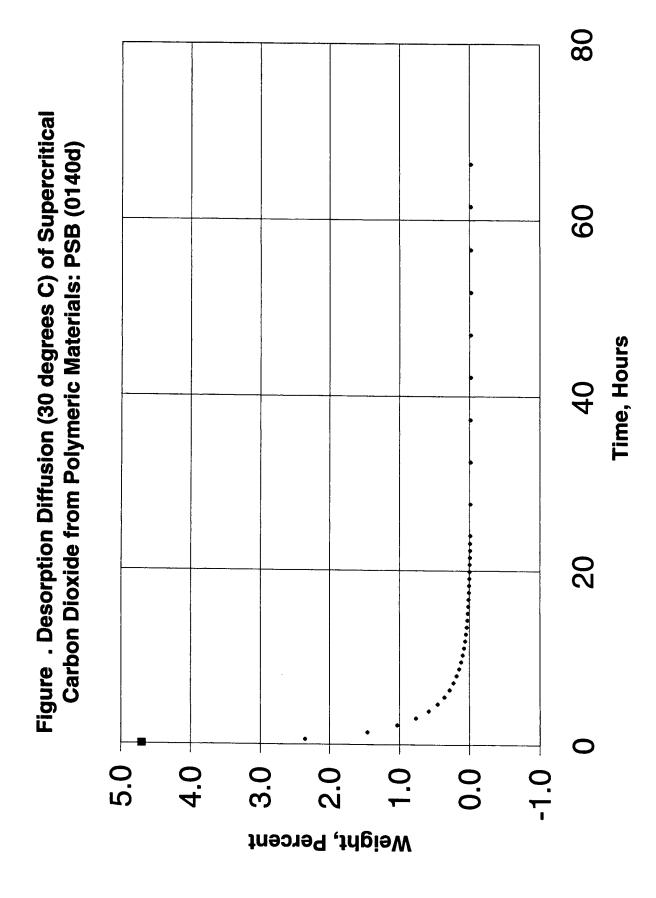
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APPENDIX B



APPENDIX B

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	Hardness	Scale	z	Shore D	!	Pre Exposure	Hardness	63.6		Post Desorption	Hardness	45.8		Change in	Hardness, %	-27.99														140 FT:40:40	01.01.13.61											
		TGA Time, sec	Z	610	,	ď.	_			Pos	_				Ĩ											4	140D			707 TO T T T T T T T T T T T T T T T T T T	Comment .384"X.075",HP @ 1400X50,Dry:DS142.0 M-142.17 1G142.018 E1.10.10											
	TGA Dry	Wt., mg	_	142.019			Data	uns	inning with n=4		•										140d.01						-BUTADIENE)		mal		red:ynd;oexor											
	Wt. @ Zero	Time, %	¥	4.704			for Analysis of (	rs refer to colun	ers to row, begi	***************************************	٠		_	_	/L4) • 100)	(74 - 14)	/ L4) * 100)	N8) /N8) - 100)			A data file : psb	14 lines in the parameter block	data			uminum Pans	JLY(STYRENE	<b>5</b>	PTION-Isother		x.075",HP <b>G</b> 14		( ) !!	ire (aC)	6		_					
	Wt. @ Zero	Time, mg	7	148.70			Formulae Used for Analysis of Data	Uppercase letters refer to columns	Lowercase n refers to row, beginning with n≖4	***************************************	On = (An * 60)	En = (Dn + M4)	Fn = (En / 3600)	Gn = SQRT (En)	Hr = (((Cn - L4) / L4) * 100)	in = ((Cn - L4) / (J4 - L4))	K4 = (((J4 - L4) / L4) * 100)	N16 = (((N12 - N8) /N8) * 100)			Source binary TA data file: psb140d.01	14 lines in the p	3 channels of data		Run 43	Module TGA Aluminum Pans	Sample PSB POLY(STYRENE-BUTADIENE) 140D	Size 146.740 mg	Method DESORPTION-Isothermal	Operator ws	Comment 384	Nsig 3	Sign time (min)	Sig2 Temperature (#C)	Sigs Weignt (mg)	Kcell 1.0000	Date 19-May-00	Time 11:49				
	Fractional	Wt. Loss	_	0.501	0.311	0.220	_	0.124	960.0	0.076	090.0	0.048		_	0.025		_		0.010	0.008	900.0	0.005	0.003	0.002	0.001								0.002	500	3	0.00	6.00 40	0.004	-0.004	-0.004 4	-0.005	900
•	Wt. Loss,	%	I	2.357	1.462	1.033	0.766	0.584	0.453	0.358	0.284	0.227	0.183	0.148	0.120	0.097	0.078	0.083	0.049	0.039	0:030	0.023	0.016	0.011	0.007	0.0	0.00	-0.003	-0.005	0.00	-0.008	0.010	6.0	0.015	-0.01	-0.018	-0.018	-0.019	-0.020	-0.02 13	20.0 P	0.02
	SORT Adj. Wt. Loss,	Time, sec	g	45.33	70.40	88.66	103.74	116.90	128.71	139.54	149.58	158.99	167.87	176.30	184.35	192.06	199.48	206.62	213.54	220.24	226.74	233.05	239.20	245.21	251.05	256.77	262.35	267.83	273.20	278.46	283.63	288.70	284.30	315.67	342.13	366.68	389.68	411.40	432.03	451.72	470.59	488.73
	Adjusted	Time, hr	u.	0.571	1.377	2.183	2.990	3.796	4.602	5.409	6.215	7.022	7.828	8.634	9.441	10.247	11.053	11.859	12.667	13.473	14.280	15.087	15.894	16.702	17.508	18.314	19.119	19.926	20.733	21.539	22.346	23.152	24.059	27.681	32.514	37.348	42.181	47.014	51.848	56.681	61.514	66.348
	Adjusted	Time, sec	ш	2055.100	4956.100	7859.740	10762.300	13664.500	16567.380	19471.420	22374.940	25278.460	28180.420	31082.800	33986.620	36888.520	39790.420	42693.460	45599.740	48504.520	51409.240	54312.640	57218.800	60125.860	63028.240	65928.940	68830.000	71733.760	74639.680	77541.760	80445.220	83348.260	86613.760	99650.020	117050.800	134451.160	151851.280	169251.460	186652.060	204052.060	221452.180	238854.100
	TGA Time,	360	۵	1445.100	4346.100	7249.740	10152.300	13054.500	15957.360	18861.420	21764.940	24668.460	27570.420	30472.800	33376.620	36278.520	39180.420	42083.460	44989.740	47894.520	50799.240	53702.640	56608.800	59515.860	62418.240	65318.940	68220.000	71123.760	74029.680	76931.760	79835.220	82738.260	86003.760	99040.020	116440.800	133841.160	151241.280	168641.460	186042.060	203442.060	220842.180	238244.100
	TGA	Wt., mg	Ö	145.366	144.096	143.486	143.107	142.848	142.663	142.527	142.422	142.342	142.279	142.229	142.189	142.157	142.130	142.108	142.089	142.075	142.062	142.051	142.042	142.035	142.029	142.024	142.019	142.015	142.012	142.009	142.007	142.005	142.003	141.998	141.995	141.994	141.993	141.992	141.990	141.989	141.988	141.988
	Temp.,	Opp	œ	30.00	30.000	30.000	30.000	30.001	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30,000	30.000	30.000	30.000	30.000	30.000	30.000	30.000	30.00	30.001	30.000	30.000	30.001	30.000	30.001	30.001	30.001	30.00	30.000	30.001	30.001	30.000	30.000	30.000	30.00	30.000
	TGA Time,	Ë	<	24,085	72.435	120.829	169.205	217.575	265,956	314,357	362.749	411.141	459.507	507.880	556.277	604.642	653.007	701.391	749.829	798.242	846.654	895.044	943.480	991,931	1040.304	1088.649	1137.000	1185.396	1233.828	1282.198	1330.587	1378.971	1433.396	1650.667	1940.680	2230.686	2520.688	2810.691	3100.701	3390.701	3680.703	3970.735